THE VISCOSITY OF STEAM AT SUPERCRITICAL TEMPERATURES AND PRESSURES

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Submitted for the Degree of Ph.D.

The University of Glasgow. December, 1960.

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PREFACE

This thesis forms a record of the experimental work carried out by the author on the measurement of the kinematic viscosity of steam in the pressure range, 200 - 800 $Kg/cm.^2$, and in the temperature range 370 - 650°C. This work was carried out on a closed circuit capillary viscometer, designed by E. Kjelland-Fosterud who carried out some preliminary work and described it in a thesis and in a published paper. The author describes the modifications which he has made to the equipment and which have enabled him to measure the kinematic viscosity with a standard deviation of ± 1.5%.(141, 142) The experimental results for steam are supported by some experiments in which the apparatus was used to measure the dynamic viscosity of nitrogen over the pressure range 10 - 117 $Kg/cm.^2$ and at temperatures around 20°C and 327°C. The experiments, using nitrogen, are particularly useful since they serve as an independent check of the values obtained for steam, and also show that an apparatus of the type described is suitable for measuring the viscosity of gases at high pressures and temperatures.

In addition to the experimental work, outlined above, the author has designed a rotating cylinder viscometer, which is described in the last chapter of this thesis. Although the closed circuit capillary viscometer has enabled a wide range of

experimental results to be obtained, the instrument is a secondary apparatus and the development of a primary instrument for the measurement of water and steam viscosities seemed desirable. In its first form, the rotating cylinder viscometer is intended for use at room temperature and pressures up to 500 Kg/cm^2, but is capable of modification for use at higher temperatures. It is estimated that this viscometer should be capable of yielding results with an accuracy of $\pm$ 0.1%.

LIST OF SYMBOLS.

Unless otherwise defined in the text, the symbols used in this thesis have the following meanings:-

a	Surface tension drag on pellet.
C	Sutherland constant.
d	Inside diameter of capillary.
D	Inside diameter of drop tube.
g	Acceleration due to gravity.
H	End correction - drop tube in vertical position.
H'	End correction - drop tube in angled position.
I	Moment of inertia.
K	Calibration constant.
ℓ	Length of capillary.
L	Distance between drop tube contacts.
m	End correction constant.
p	Pressure.
Q	Volume flow.
r	Inside radius of capillary.
Re	Reynolds Number.
t	Time.
T	Temperature.
v	Specific volume.
V	Linear velocity.

w Mass flow.

W Mass of mercury pellet.

δ Logarithmic decrement.

δ_0 Logarithmic decrement in vacuum.

Δ Standard deviation of timings - drop tube in vertical position.

Δ' Standard deviation of timings - drop tube in angled position.

η Dynamic viscosity.

λ Mean free path.

ρ Density.

ν Kinematic viscosity.

LIST OF FIGURES.

INTRODUCTION

The viscosity of a fluid represents the tendency for adjacent layers of the fluid flowing with different velocities to assume the same velocity. This is effected by a transfer of momentum from one layer to the next, and the measure of this transfer of momentum is the measure of the viscosity. The associated transport properties of the fluid - conductivity and diffusivity - are due to the corresponding transfer of energy and of the molecules themselves.

A general equation for viscosity was derived by Maxwell[1]* and may be written in the form

$$\gamma = \frac{1}{3} \rho \bar{v} \lambda \qquad \text{.....(1)}$$

where λ denotes the mean free path of the molecules and $\bar{v}$ their mean velocity. The assumptions used to formulate this equation are those of the ideal case of a gas whose molecules are spherical and do not exert attractive forces. The effect of molecules moving with finite velocity after collision and the distribution of velocities along the mean free path are neglected. For an elastic spherical molecule Chapman[2] and Enskog[3] have modified the constant to 0.449 to take account of the former and Boltzmann[4] has shown that the constant becomes 0.35, if correction

* Numbers in brackets above line refer to the bibliography on page 145 .

is made for the latter. In all cases the mean free path is assumed constant for all the molecules and may be given by the expression

$$\lambda = \frac{m}{\sqrt{2} \cdot \pi \cdot \sigma^2} \qquad \text{.....(2)}$$

where m and σ are the molecular mass and diameter respectively. In equations (1) and (2) it is possible to obtain a numerical value for the molecular velocity, $\bar{v}$, and for the molecular mass, m, but no accurate values are available for the molecular diameter, σ, other than these derived from measured values of viscosity. A satisfactory numerical value of viscosity cannot, therefore, be obtained from equations (1) and (2). In the case of water or steam, where the molecules are not true elastic spheres and where the further complication of polarity is introduced, it is impossible to obtain an accurate value of viscosity from these equations.

Several equations are available, however, which are based on kinetic theory and which can be used to predict the effect of pressure and temperature on viscosity. These equations are not suitable for absolute determinations of viscosity since they contain constants whose values can only be obtained by substituting known values of viscosity into the equations. Certain predictions may also be made from equations (1) and (2).

From equation (1) it may be seen that viscosity is independent of pressure since the mean molecular velocity is independent of pressure, density is directly proportional to pressure and the mean free path is inversely proportional to

pressure. This has been found to be true for steam except at high pressures where the length of the mean free path of the molecules tends to be of the same order of magnitude as the molecular diameter, and at low pressures - below $^{1}/_{60}$ atmospheres according to Meyer[5] - where the number of molecules in the system is relatively small and the length of the mean free path tends to be of the same order of magnitude as the dimensions of the system. A prediction which fits the facts more closely at high densities may be made by treating the molecules as rigid spheres using the equations of Enskog, Boltzmann and others but the evaluation of the constants in these equations again requires experimental viscosity results. A recent comparison of the Enskog theory with experimental data has shown discrepancies of more than 50% for steam[6] and the theory can, therefore, be used only as a guide.

The temperature dependence of viscosity may also be predicted from equations (1) and (2). Since the molecular velocity is proportional to the square root of the absolute temperature for elastic spherical molecules, the viscosity is also proportional to the square root of the temperature. Experiment has shown, however, that the dynamic viscosity is proportional to the temperature raised to a power which is greater than $\frac{1}{2}$ in all cases. A more satisfactory equation for deriving the temperature dependence of viscosity is that given by Sutherland[7]

in the form

$$\eta = \frac{a\sqrt{T}}{1 + \frac{C}{T}} \qquad \ldots\ldots\ldots\ldots(3)$$

where a and C are constants. This expression gives satisfactory results for a large number of gases over limited ranges of temperature but the evaluation of the constants depends on experimental values of viscosity. A refinement of this expression has been given by Lennard-Jones[8] in the form

$$\eta = \eta_o\left[\frac{T}{T_o}\right]^{\frac{3}{2}} \frac{C + T_o}{C + T}^{\frac{\nu-3}{\nu-1}} \qquad \ldots\ldots\ldots\ldots(4)$$

where ν depends on the interaction potential and the index 3 is adopted as a mathematical convenience. This equation takes into account the "softness" of the molecules and their mutual attraction but also requires experimental viscosity values to determine the constants.

Hirschfelder, Bird and Spotz[9] have stated that the above equations cannot be used to give accurate viscosity results for steam. Additional assumptions are necessary to take account of the polarity of the molecules and this will result in the formidable task ofevaluating collision integrals.

It is apparent from the consideration of equations based on the kinetic theory of gases that the viscosity of water and steam cannot be accurately predicted from this source. The viscosity

must, therefore, be evaluated from hydrodynamic theory.

The normal hydrodynamic definition of viscosity is that given by Newton's hypothesis which states that

$$\tau = \eta \frac{du}{dy} \qquad \ldots\ldots\ldots\ldots(5)$$

where τ is the shearing stress between adjacent parallel planes of a fluid of dynamic viscosity η, in steady flow. The fluid flows with velocity u parallel to a fixed plane and velocity differences occur in the y direction, normal to the wall. This definition is incomplete since it applies only to steady, laminar flow and does not apply for low rates of shear. The complete hydrodynamic definition is given by the Navier - Stokes equations which are the fundamental equations of hydrodynamics and which may be written in the form

$$\left.\begin{aligned}
\rho \frac{Du}{Dt} &= X - \frac{\partial p}{\partial x} + \eta \nabla^2 u + (\eta + \eta') \frac{\partial}{\partial x}\left(\frac{\partial u}{\partial x} + \frac{\partial v}{\partial y} + \frac{\partial w}{\partial z}\right) \\
\rho \frac{Dv}{Dt} &= Y - \frac{\partial p}{\partial y} + \eta \nabla^2 v + (\eta + \eta') \frac{\partial}{\partial y}\left(\frac{\partial u}{\partial x} + \frac{\partial v}{\partial y} + \frac{\partial w}{\partial z}\right) \\
\rho \frac{Dw}{Dt} &= Z - \frac{\partial p}{\partial z} + \eta \nabla^2 w + (\eta + \eta') \frac{\partial}{\partial z}\left(\frac{\partial u}{\partial x} + \frac{\partial v}{\partial y} + \frac{\partial w}{\partial z}\right)
\end{aligned}\right\} \ldots(6)$$

In this expression η represents the first coefficient of viscosity of the fluid, or shear viscosity, and η' the second coefficient of viscosity or dilatation viscosity. The equations represent the relationship between the principal stresses and the principal rates of deformation at any point in any homogeneous fluid. It

was derived independently by Navier[10], based on intermolecular forces, and by Stokes[11] based on Newton's hypothesis.

The Navier - Stokes equations may be simplified for the particular case of unidirectional, steady flow to give

$$\frac{\partial p}{\partial x} = \gamma \frac{\partial^2 u}{\partial y^2} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (7)$$

which equation may be integrated with suitable boundary conditions to give an expression involving pressure drop, velocity, distance and viscosity. It remains, therefore, to devise an instrument with boundary conditions to suit the above equation in which the pressure drop, velocity and distance are easily measured.

CHAPTER I

METHODS OF MEASURING THE VISCOSITY OF NEWTONIAN FLUIDS.

1.1 TRANSPIRATION

The transpiration method of measuring the viscosity of a fluid is based on the application of the Hagen-Poiseuille equation to the laminar flow of a fluid through a straight tube of circular cross-section. The equation was derived independently by Hagen[12] and Poiseuille[13] from their experiments. It may be derived from the Navier - Stokes equation in the following manner:-

Consider a fluid flowing with steady unidirectional flow along a circular tube of radius R. The velocity components in the tangential and radial directions are zero and the velocity u along the pipe in direction x depends only upon y, the radial component measured from the axis outwards. The pressure is constant at every cross-section and the Navier - Stokes equation, in cylindrical coordinates, reduces to

$$\frac{d^2u}{dy^2} + \frac{1}{y}\frac{du}{dy} = \frac{1}{\eta}\frac{dp}{dx}$$

$$\therefore \quad \frac{d}{dy}\left(y \cdot \frac{du}{dy}\right) = \frac{y}{\eta}\frac{dp}{dx}$$

$$\therefore \quad y\frac{du}{dy} = \frac{y^2}{2\eta} \cdot \frac{dp}{dx} + C'$$

assuming that there is no slip, the boundary conditions are

$$\frac{du}{dy} = 0 \text{ at } y = 0 \text{ and } u = 0 \text{ at } y = R.$$

$$\therefore \quad u = -\frac{1}{4\eta} \frac{dp}{dx} (R^2 - y^2)$$

$$\therefore \quad u_{max} = \frac{1}{4\eta} \cdot \frac{dp}{dx} R^2.$$

Since the velocity across the cross-section is distributed in the form of a parabaloid of revolution the mean velocity $\bar{u}$ is given by $\frac{u_{max}}{2}$

$$\therefore \quad \bar{u} = -\frac{R^2}{8\eta} \frac{dp}{dx}$$

$$\therefore \quad \eta = -\frac{\pi R^4}{8Q} \frac{dp}{dx} \quad \ldots\ldots\ldots \text{Hagen-Poiseuille equation,}$$

where Q is the volume flow rate.

Hagen also postulated that the pressure drop across the tube was composed of two terms, one involving mass flow and the other involving mass flow squared. He suggested that the first term represented the pressure head required to overcome friction and the second term represented the pressure head required to supply kinetic energy.

The limits of validity of the Hagen-Poiseuille equation are discussed in Chapter 4, with particular reference to the apparatus used to determine the viscosity of steam. Equations of a similar type have been derived by Meyer[14], Fisher[15] and Trautz and Weizel[16] and a simple derivation of the above

equation from Newton's hypothesis is given by Prandtl[17].

The application of the equation to the measurement of the viscosity of fluids has been attempted using many different forms of apparatus. Two general types are available for the measurement of the viscosity of steam.

(a) Open Circuit Viscometer.

In this type of viscometer steam is supplied to the capillary from an external source. The steam then passes through the capillary at constant temperature, is throttled by means of a valve, condensed and weighed. The pressure drop across the capillary is measured by means of a manometer.

To ensure that the viscosity determination may be of reasonable accuracy a large flow rate is required since this tends to minimise any errors due to pump fluctuations or desnity fluctuations from other sources. It also permits the use of a larger capillary bore diameter while retaining a measurable pressure drop. The same pressure drop may be obtained in two ways, for a given flow rate, and each gives rise to its own problems. Since the pressure drop is proportional to the capillary length and inversely proportional to the radius raised to the fourth power, we may have

(i) a capillary of considerable length with an internal diameter such that it can be accurately manufactured and measured. This gives rise to a primary instrument

suitable for absolute determinations of viscosity. The instrument also has a large length to diameter ratio which effectively minimises any end effects due to the non-parabolic velocity distribution at the entrance to and exit from the capillary.

It is necessary to maintain the temperature of the steam constant along the length of the capillary. Using a long capillary - say one hundred feet - this becomes a practical problem of considerable magnitude. Some experimenters have partly overcome this problem by coiling the capillary and attempting to maintain the resulting toroid at a constant temperature. This technique, however, necessitates the use of a correction to allow for the effect of coiling, a correction whose value may be in some doubt. Hawkins[18] found for his coiled capillary that the viscosity became dependent upon the value of the Reynolds number for a Reynolds number greater than 180. This necessitates the calibration of the apparatus for all values of Reynolds number above 200 and it ceases to be a primary instrument; and (ii) a relatively short capillary with a bore which cannot be accurately measured. Techniques involving X-rays or electrical probes can be used with a known mass of mercury to measure the diameter of the capillary provided

the diameter is reasonably constant. These methods have been successfully used for mercury manometers and experiments have shown that with a suitable form of X-rays it is possible to measure bore diameters of 0.5 mm. to within 0.002 mm. If the diameter is too small for these techniques or if the diameter varies along the length, the capillary may be calibrated against a fluid of known viscosity. The instrument is then a secondary viscometer.

A correction should be applied to account for the pressure loss due to the flow configuration at the ends of the capillary, but the steam temperature along the capillary is more readily controlled than in (i).

The problems of steam production and pressure measurement are common to both forms of open circuit viscometer. The comparatively large flows required, necessitate the use of a steam generator to produce the steam at working pressures and temperatures of 800 $Kg/cm.^2$ and $700^{\circ}C$. Such a generator would necessitate a major research programme. The manometer required to measure the pressure drop across the capillary must also be subject to the total working pressure and this renders visual readings impossible although the manometer may be at room temperature. Differential pressure measurement must, therefore,

be by X-ray methods, inductance or capacitance pick up, or by electrical probes. Considerable care must be taken to ensure that the limbs of the manometer are horizontal from the capillary ends to the steam-water interface, otherwise buoyancy errors are introduced. Alternatively the manometer may be situated in the steam space, an arrangement which avoids the buoyancy correction but introduces design difficulties since the manometer must be read at some temperature above the critical temperature for water.

(b) Closed Circuit Viscometer.

This is an adaptation of the Rankine[19] viscometer, in which the gravitational displacement of a known mass of mercury creates the pressure difference across the capillary. The mercury displaces the fluid under conditions of known dimensions and the volume flow rate is therefore known. The method of displacement of the fluid used by Rankine is simple and takes the form of a mercury pellet falling in a straight glass tube of known diameter. The time of fall is measured for a known length.

This technique may be easily applied to the measurement of the viscosity of steam, the capillary being situated in a furnace at the steam test temperature and the tube containing the

mercury located outside the furnace at room temperature. The capillary for this form of apparatus must have dimensions such that a measurable volume displacement is produced by a mercury pellet of reasonable dimensions. An approximate evaluation of the Hagen-Poiseuille equation shows that the capillary for this apparatus has an internal diameter so small that it cannot be accurately measured and the apparatus must, therefore, be calibrated against a fluid of known viscosity. A closed circuit viscometer for use with steam is, therefore, a secondary instrument.

1.2 ROTATION.

The rotational method of measuring the viscosity of a fluid depends on the principle that a solid object suspended in a fluid, such that it is completely immersed, will be displaced if subjected to a viscous drag. This viscous drag is normally created by the rotation, at constant speed, of a second object of similar shape which is co-axial with but larger than the suspended object. The displacement of the suspended object is a measure of the viscosity of the fluid.

All accurate determinations use a torsion wire as the means of suspending the body in the fluid. The geometrical shape of the suspended and rotated bodies generally takes the form of a sphere, a disc or a cylinder, and the theory related to each of these shapes is now discussed.

(a) Sphere. Theoretically this represents the ideal

rotational method of measuring the viscosity of a fluid. Brillouin[20] showed that for the case of a fluid rotating between concentric spheres the viscous drag on the suspended sphere of radius b is given by

$$G = -8\pi\gamma\Omega\frac{a^3 b^3}{b^3 - a^3}$$

where the sphere of radius a rotates with constant angular velocity Ω in a fluid of dynamic viscosity γ.

In spite of its theoretical completeness few experimeters have used this method, since the practical difficulties render its construction virtually impossible. Only Zemplen[21] attempted to overcome the difficulties with any measure of success and yet his results for air are considerably higher than the accepted values.

(b) Disc. Of the three simple geometric forms, the theory pertinent to the rotating disc is the most difficult to predict. The disc is not, therefore, used for absolute determinations of viscosity. It has, however, proved useful for the measurement of large viscosities relative to a fluid of known viscosity.

For the ideal case of an infinitely thin disc rotating in an infinite fluid with angular velocity Ω, the retarding couple may be shown to be

$$G = -\frac{32}{3}\gamma\Omega a^3$$

where a is the radius of the disc.

The alternative case of the disc rotating between fixed, parallel planes, each at a distance h from it gives

$$G = -\pi \eta \Omega \frac{a^4}{h}$$

where a >> h.

Both formulae are approximate, in a practical application, since the disc must have an edge of finite thickness and a suitable correction for the effect of this edge is not accurately determinable. The flow for the above formulae must be purely circumferential but it would appear that radial flow is unavoidable.

(c) Cylinder. Maragules[22] derived the theory for the case of a cylinder of infinite length and radius a rotating with a fixed angular velocity Ω, inside a second infinite cylinder of radius b, which is fixed. He showed that the retarding couple is given by

$$G = 4\pi \eta h \Omega \left[\frac{b^2 - a^2}{a^2 b^2}\right]$$

where h is the length of the inner cylinder which is under consideration. He assumed that the flow of the viscous fluid was laminar and entirely circumferential between the two cylinders and that there was no slip. The equation may be derived from the Navier-Stokes

equation in the manner shown by Schlichting[23].

With the above assumptions, the theory is equally valid for the case with either the inner or outer cylinder rotating.

Two unknown quantities arise from this evaluation -

(i) The conditions necessary to guarantee the laminar flow required by the assumptions.

(ii) The effect of the cylinders not being of infinite length.

The problem of laminar flow has been treated theoretically by Rayleigh[24] and Taylor[25], and experimentally by Couette[26], Mallock[27] and Taylor. Rayleigh predicted that at low speeds, all steady motions of a viscous fluid must be stable and that the critical Reynolds number* for the outer cylinder rotating would be approximately 2,100 and for the inner cylinder rotating would be considerably less. Couette's experiments - using an apparatus in which the outer cylinder rotated - showed close agreement with these predictions. Couette determined the critical Reynolds number to be 1900. Mallock succeeded in maintaining laminer flow at Reynolds numbers up to the range 4 to 32 x 10^3 but found instability at all speeds for the case where the inner cylinder rotated.

* Reynolds number for this case may be written

$$Re = \frac{\Omega a (b - a)}{\nu}$$

The more comprehensive work of Taylor suggests that Couette and Mallock were in error and that laminar flow exists for all speeds of the rotating outer cylinder. His apparatus running at its maximum speed - corresponding to a Reynolds number of 12,500 - showed no sign of instability. For the case of the inner cylinder rotating, he proved that the critical speed is given by the equation

$$\frac{\pi^4 \nu^2 (a + b)}{2 \Omega^2 t^3 b^2} = 0.0571\left(1 - 0.652 \frac{t}{b}\right) + 0.00056 \left(1 - 0.652 \frac{t}{b}\right)^{-1}$$

where $t = (b - a)$, and it is assumed that $\frac{t}{a}$ is small.

In a subsequent paper, Taylor[28] suggested that there is a lower critical value of Reynolds number below which the flow is at all times laminar - 1800 - and an upper critical value above which the flow is at all times turbulent. At intermediate points the flow is normally stable but a slight disturbance is sufficient to render the flow entirely unstable. For the case of the inner cylinder rotating he applied, as a first approximation $\frac{t}{b} \to 0$ and proved, from his previous formula, that

$$\left[\log \frac{\Omega}{\nu}\right]_{\text{critical}} = 0.818 - \frac{1}{2} \log a. - \frac{3}{2} \log t.$$

It may be seen from these investigations that, although they do not agree exactly, the ratio of critical Reynolds number for the case of the outer cylinder rotating to that of the inner cylinder rotating is of the order of 50 to 1.

The given formula for the viscosity relationship does not take into account the effect of the finite length of the cylinders. No satisfactory correction has been theoretically evaluated for this source of error but it is possible to render the necessary correction a minimum by different means. The method originally used by Couette is the most practical and is the only one considered here.

This entails the positioning of two cylinders, of exactly the same diameter as the suspended cylinder, immediately above and below the suspended cylinder and concentric with it. The length of the rotating cylinder is then the sum of the lengths of these guard cylinders, the suspended cylinder and the gaps between the guard cylinders and the suspended cylinder. A small correction is required to allow for the effect of these gaps since the total torque output must equal that transferred to the suspended cylinder, the guard cylinder and to the sum of the gaps.

The torque on the gaps may be said to be equally shared between the guard cylinders and the suspended cylinder, i.e. the effective length of the suspended cylinder becomes the actual length plus 2 x $(\frac{1}{2})$ gap. This argument is not entirely justified since at the gap, the effective distance between the rotating and suspended cylinder is greater than elsewhere and the correction

to the effective length of the suspended cylinder should, in consequence, be slightly smaller than that suggested above. The total correction may be arranged to represent approximately 0.1% of the value of the viscosity and the slight error inherent in the assumption, may be neglected.

It appears, therefore, that the rotating cylinder is suitable for measurements of the viscosity of steam provided the design problems, associated with the high temperatures and pressures, can be overcome.

1.3 FALLING BODIES.

This method of measuring viscosity is based on Newton's statement that when a body is acted upon by a constant force, and resistance to its motion is proportional to the velocity or to any power of the velocity, the body will eventually attain a constant or terminal velocity, such that the resistance experienced is equal to the driving force.

Stokes[29] developed the following law for the particular case of a sphere moving with uniform velocity

$$W = 6\pi\eta a. v.$$

where W is the resistance to motion, a the radius of the sphere and v the constant velocity. From this equation it is easily shown that for a sphere falling under the action of gravity in a viscous fluid

$$\eta = \frac{2}{9} \frac{(\sigma - \rho)}{v} . g. a^2$$

where σ is the density of the sphere and ρ the density of the fluid.

This law is subject to the following assumptions:-

(i) the motion of the sphere relative to the fluid is slow, steady and free from acceleration,

(ii) the fluid is homogeneous and of infinite extent,

(iii) the sphere is rigid,

(iv) no slip takes place between the sphere and the fluid.

The first assumption implies that the flow past the falling body is laminar and of constant velocity. Rayleigh has shown that this is only true if $Re < 1$ and subsequent experimental investigations have shown that it is desirable to have $Re \ll 1$. The second assumption necessitates a fluid of infinite extent which is not practically possible. The third and fourth assumptions are readily satisfied, but the first two cannot be satisfied with sufficient accuracy to justify the use of the apparatus for absolute measurements.

The method can, however, be used for relative measurements where the assumptions need not be satisfied to the same extent. For relative measurements the fluid need not be of infinite extent but a correction must be applied for the effect of the ends of the containing tube. The available corrections are empirical and their accuracy is in some doubt.

It has been shown by Barr[30] that the ratio of ball

diameter to the tube diameter cannot be greater than 1:20 if an accuracy of $\frac{1}{2}\%$ is to be obtained from an absolute determination. Clearly this ratio could not be used for water or steam and a fall tube diameter very slightly longer than that of the sphere would be required. This would increase the Reynolds number considerably and cause turbulence.

Hawkins[31] experimented with different shapes of falling body in an attempt to retard the fall of the body and maintain the Reynolds number below the critical. Their experiments were not successful. Sage and Lacey[32], Schroer and Becker[33], Smith and Brown[34] and others have attempted the measurement of the viscosity of gases using the falling body method. The results over the greater part of the range are in error by more than 5%. The method has, however, been applied to liquids with a reasonable degree of success by Fritz and Weber[35].

1.4 OSCILLATION.

The oscillation viscometer is mechanically the simplest possible and consists of a mass of known moment of inertia, I, performing torsional oscillations on the end of a suspension - normally a wire. The system is completely immersed in a viscous fluid and the motion of the mass is then defined by the equation

$$I \frac{d^2\alpha}{dt^2} + L \frac{d\alpha}{dt} + M\alpha = 0$$

where $-L \frac{d\alpha}{dt}$ is the moment of the viscous force and $M\alpha$ is the couple required to displace the suspension through the angle α, in

the absence of any viscous force. The solution of the equation is

$$\alpha = A e^{-\delta \frac{t}{T}} \cos. 2\pi \frac{t}{T}$$

where α is the angular displacement at a time t, T is the period of the oscillation and δ the logarithmic decrement of the amplitude for one complete oscillation. This general theory has been applied to a number of simple geometric shapes and, as in the case of the rotation viscometer, the sphere, the cylinder and the disc are discussed.

(a) Sphere. The sphere may be used in the following forms to measure the viscosity of a fluid.

(i) The viscous fluid outside a solid sphere and extending to infinity.

(ii) The viscous fluid outside a solid sphere and bounded by a concentric sphere.

(iii) The viscous fluid outside a hollow sphere and extending to infinity. The same fluid, or another, fills the interior of the sphere.

(iv) The viscous fluid outside a hollow sphere and bounded by a concentric sphere. The same fluid, or another, fills the interior of the sphere.

(v) The viscous fluid inside a hollow sphere with vacuum outside.

For each of the above cases, there exists the problem of constructing a sphere, or spheres, to a suitable accuracy.

Also the theory evaluated for each of the above methods cannot be applied with sufficient accuracy, particularly to gases where due to the low dynamic viscosity the damping is small. A complete investigation into the case of the oscillating sphere has been carried out by Kestin and Person[36] and in a later publication Kestin and Wang[37] assessed the applicability of oscillating spheres to the measurement of gas viscosity. They concluded that it may be possible to use a small sphere at high viscosities provided it could be manufactured with sufficient accuracy.

Moszynski[38,39] - see also Kestin[40,41] - has successfully used a solid stainless steel sphere, 1.1251 in. ± 0.0001 in. diameter, to measure the viscosity of water at temperatures up to 186°C. and at pressures up to 341 atmospheres. The measured points agree closely with other authors and do not deviate from a smooth curve by more than 0.05%. Moszynski concluded that his experimental accuracy was better than ± 0.1% and his absolute accuracy ± 1%.

The same pressure vessel and equipment, with the exception of the oscillating system, were used by the same author for his measurements on steam. Moszynski did not attempt to use the oscillating sphere for his measurements on steam but introduced a new system with a disc as the oscillating body.

(b) Cylinder. A theoretical treatment of the solid - or hollow - cylinder performing torsional oscillations has been given by Kestin and Person[36]. The solutions given by them are exact solutions of the Navier-Stokes equation, the inertia terms being neglected. This effectively limits the theory to small oscillations such that secondary motion is negligible.

A satisfactory correction for the effect of the cylinder ends has not been found and this, together with the difficulty of eliminating secondary flows, rules out the use of the oscillating cylinder as a method of measuring viscosity.

(c) Disc. The oscillating disc may be applied to the measurement of viscosity in two forms and these are now discussed in detail.

(i) Oscillation in an infinite fluid. The first approximate analytical solution for this case was obtained by Meyer[42] and was based on the assumption that the viscous fluid forms a cylinder of which the disc is part and that the effect of the surrounding fluid is neglected. His equation may be written as

$$\delta = \frac{\pi a^4 \sqrt{\pi \rho \eta T}}{2\sqrt{2}\, I} + \frac{\pi a^3 \eta T}{I}$$

where T is the period of oscillation, I the moment of inertia of the oscillating system and a the radius

of the disc. The second term of the equation represents the effect of the edge. Improvements have been made to this equation by Verschaffelt[43] and Kobayaski[44] who corrected for the effect of the moment of inertia of the fluid itself. Subsequent experiments using the improved forms of Meyer's equation have not yielded consistent results.

Further theoretical improvements have been made by Macwood[45] and Hollis-Hallet[46] who used an equation of the form

$$\eta = k \eta_0$$

where k is the correction factor for the edge effect and η_0 the uncorrected viscosity. The expression for η_0 given by both authors is essentially the same and both made the assumption that $\delta^2 \ll 1$. Results based on these theories have again not yielded consistent results - probably due to the inaccuracy of the edge corrections.

Theories have also been advanced by Kestin and Person[47] and by Mariens and Von Paemel[48]. The former authors evaluated the ideal case of a body with smooth boundaries and later[37] introduced practical corrections for wire damping, side drag and drag on the rod carrying the mirror. This report also introduced an edge correction factor but since for infinite spacing the correction is likely to be of the order of 30% of the drag on the

disc, it cannot be accurately applied to experiment. Mariens and Von Paemel applied their theoretical interpretation to experiment and found that the results deviated by as much as + 7.1% for air and - 3.9% for water.

Recently Appeitia and Newell[49,50] analysed the problem for the thin disc and the thick disc, i.e. the boundary layer thickness large compared with the disc thickness and small compared with the radius, and the boundary layer small compared with both the disc thickness and its radius respectively. The solution of the equations derived for each case is achieved by a process of successive approximation - probably most effectively carried out with the aid of a digital computer - and is subject to an estimated error of $\pm$ 0.5%. Recalculation of the experimental data of Kestin and Pilarczyk[51] and Kestin and Wang[52] is claimed to substantiate this accuracy and appears to do so. The geometrical limitations placed on this theory by the assumptions also limit the range of values of viscosity for which the theory is valid.

(ii) Oscillation between two fixed plates. A solution for the finite disc oscillating between two fixed plates was obtained by Maxwell to evaluate his measurements of the viscosity of air. The solution is based on the assumptions that each part of the disc which is not near the edge should be treated as part of an infinite disc and that no radial flow exists.

A more refined theory was presented by Macwood[45] who evaluated the viscous damping on the surface of an infinite oscillating cylinder of the same radius as the disc. Assuming that the cylinder had a length equal to the disc thickness, 2d say, he obtained the expressions.

Small Separation - $$\eta = \frac{2 I x}{\pi a^4 T}\left[\left(\frac{T}{T_o}\right)^2 \frac{\delta}{2\pi} - \left(\frac{T}{T_o}\right)\frac{\delta_o}{2\pi}\right](1 - E)$$

where $$E = \frac{8 d x \sqrt{\frac{\pi\rho}{T\eta_1}}}{4 d x \sqrt{\frac{\pi\rho}{T\eta_1}} - a}$$

Large Separation - $$\eta = \left\{\frac{4 I}{\pi a^4}\sqrt{\frac{\pi}{T\rho}}\left[\left(\frac{T}{T_o}\right)^2 \frac{\delta}{2\pi} - \left(\frac{T}{T_o}\right)\frac{\delta_o}{2\pi}\right]\left[1 - \frac{\delta}{4\pi}\right]\right\}^2 (1 - E)$$

where $$E = \frac{2\sqrt{\frac{T\eta}{\pi\rho}} - 4 d}{2\sqrt{\frac{T\eta}{\pi\rho}} + \left(\frac{a}{2} - 2 d\right)}$$

for both expressions it is assumed that $\delta^2 << 1$ and $\delta_o^2 << 1$. The separation between the fixed and oscillating plates is x and E is the edge correction. The period of the oscillating system is T and the corresponding period in vacuum is T_o. Similarly δ and δ_o are the logarithmic decrements of the system in the fluid andin vacuum; a is the disc radius.

The formula pertinent to a small separation has been proved by Kestin and Pilarczyk[51] to be unreliable. They applied it to an experimentally reliable apparatus and obtained a value for the viscosity of air which was 9% too high. This is probably due to the erroneous assumption made by both Maxwell and Macwood, that

the angular velocity of the fluid is constant up to the edge of the disc.

Macwood's formulae are derived from two expressions of a hyperbolic tangent and for a theoretical error of less than 1%, due to neglecting all the terms of the expansion except the first two, we must have

for a small separation $x\sqrt{\frac{\pi\rho}{T\gamma}} < 0.3$

and for a large separation $x\sqrt{\frac{\pi\rho}{T}} > 4.$

It is also necessary to ensure that the angular velocity is small since radial and axial components of velocity must be eliminated. The Reynolds number may be defined as $\frac{\Omega a^2}{\nu}$ for this case and the critical Reynolds number is that value below which centrifuging cannot exist. Cochran[53] has stated that values of Reynolds number less than 5×10^5 will ensure laminar flow. This value is not in accordance with that given by Foch and Barriol[54] whose experiments indicated that secondary motion could not exist provided $Re < 60$. The value given by Cochran was derived for an infinite plane rotating with constant angular velocity and for this ideal case is correct but for practical oscillations it is desirable to maintain $Re < 60$.

The theories advanced for the ideal case are all in agreement and only the correction for the effect of the edge and the elimination of radial flow prevent the 'finite spacing' oscillation viscometer from becoming the optimum method of measuring viscosity. The edge correction may account for 10% of the drag

in the disc and to assess it accurately Kestin and Wang[37] derived an empirical method using successive approximations. This is a secondary method of evaluating the correction since it necessitates calibration against a fluid of known viscosity. In their report, however, the authors claimed that a primary theory had been evaluated which agrees with the empirical values to better than 0.1%.

This claim has since been substantiated in a paper by Newell[55] which constituted the last of a series of fire papers concerned with oscillation type viscometers. An expression for the edge correction was evolved from first principles, the method used being similar to that for infinite spacing but with boundary conditions to suit the geometry of the system. The solution is exact for the particular case where the boundary layer thickness

$$\delta = \left[\frac{\eta}{2\pi \rho T_o} \right]^{\frac{1}{2}}$$

and the radius of the disc are large compared with the separation of the plates. This condition limits the use of the theory to low densities but nevertheless appears to be the first theory suitable for application to absolute determination of the viscosity of gases by the observation of the period of a thin disc performing small torsional oscillations between two fixed plates.

The new theory represents an extension of that given by Kestin and Person[56] for the case of an infinite disc of radius R oscillating between two fixed plates, each separated from it by a distance b. It was assumed that the disc was infinitely thin and

the characteristic equation for this case may be written as

$$(s + \Delta_0)^2 + 1 + \frac{\pi R^4 \rho \delta}{I} \cdot s^{3/2} \cdot \coth. (\beta s^{\frac{1}{2}}) = 0$$

where Δ_0 is the decrement of damping of the wire in vacuum and β the dimensionless separation $(\frac{b}{\delta})$.

Newell obtained a solution to this equation by applying a series expansion to the hyperbolic tangent. The solution is, however, very complicated and simplification results in a corresponding loss in accuracy. The theory has since been used by Kestin and Leidenfrost[57,58,59] for their measurements on gases and appears to substantiate the claimed accuracy. These authors found it necessary to employ an automatic digital computer to perform the numerical calculations involved in the evaluation of the results.

1.5 CHOICE OF METHOD

The author's choice of a method of measuring the viscosity of steam was limited by the existence of the Rankine type viscometer designed by Kjelland-Fosterud[60,61], and the immediate requirement of experimental results. Examination of the apparatus and of the results obtained by Kjelland-Fosterud showed that these results should be regarded as the results of a preliminary investigation and that the work should be repeated. It was, therefore, decided that the existing apparatus should be modified to enable reliable results to be obtained from it.

Results obtained from this apparatus are of a secondary nature, since the apparatus has to be calibrated against a fluid

of known viscosity. It is, therefore, desirable that results be obtained from an independent apparatus preferably of a primary nature. The fore-going discussion of the available methods of measuring the viscosity of Newtonian fluids indicates that the falling body viscometer is theoretically unsuitable, that the open circuit capillary viscometer and the rotating cylinder viscometer are mechanically complicated and that the oscillating disc is a secondary instrument over a large range of densities although primary results may be obtained at the later density values.

The oscillating disc appeared, mechanically, to be the most suitable instrument but at the commencement of the present work the theory derived by Newell had not been published and it appeared that the instrument was a secondary one over its entire range. Further it was known that Kestin had undertaken a full scale research programme into the measurement of the viscosity of gases - including steam - using an oscillating disc, and it seemed unnecessary to duplicate the work. Kestin has since stated[62] that he has been unable to find a suspension material suitable for temperatures in excess of 300°C.

The open circuit capillary viscometer was considered but since the existing Rankine type viscometer incorporates a capillary it was decided to investigate more closely the problems associated with a rotating cylinder viscometer. This decision was substantiated by the knowledge that Schmidt proposed to construct an instrument of the open circuit capillary type at the Technische Hochschule in Munich.

The rotating cylinder viscometer is theoretically complete although small corrections must be applied for the effect of the gap between the suspended cylinder and the guard cylinders and for the torque on the suspension, other than the cylinder itself. It has not, however, been used for measurements at temperatures above atmospheric and has been used only once[63] for measurements at pressures above atmospheric and then to a maximum pressure of only 30 Kg/cm^2

The mechanical difficulties associated with this form of apparatus appear to be the cause of this limited range. It was, therefore, decided to investigate these problems and to attempt to construct an apparatus for use at atmospheric pressure but capable of modification for use at the maximum conditions for which the rotating cylinder is suitable.

CHAPTER 2.

REVIEW OF LITERATURE.

In order to assess the accuracy of measurements made in the past it is necessary to make a critical survey of these works. This survey is conveniently divided into two parts, the first of which comprises measurements made to determine the viscosity of the possible calibrating fluids and the second comprises measurements made on water and steam.

2.1. CALIBRATING FLUIDS.

The capillary apparatus described in this report is a "secondary instrument" and must be calibrated against a fluid whose viscosity is accurately known at atmospheric conditions of temperature and pressure. A brief study of the relevant literature shows that there are two possible gases for this purpose - air and nitrogen.

Kestin stated at the 1956 Steam Conference in London that he favoured the use of Nitrogen since it can be obtained 99.999% pure and its viscosity is accurately known. Air, however, is more convenient and, although its purity may not always be as high as that of nitrogen, a considerable amount of experimental work has been directed towards the determination of its viscosity. The author, therefore, feels that a study of the literature relevant to the measurement of the viscosity of these gases is desirable, in order to establish the present accuracy of the accepted values at atmospheric pressure and temperature.

(a) Nitrogen. The only determination of the viscosity of nitrogen by the rotating cylinder method is that of Kia-Lok Yen[64] in 1919. This work was carried out on the instrument previously used by Gilchrist and Harrington and this instrument would seem to have been capable of yielding results of high accuracy. Yen claims an accuracy of 0.1% for his measurement at 23°C., - Gilchrist claimed 0.2% accuracy for his determinations of air in 1912 - and this appears to be justified.

Michels and Gibson[65] using a Rankine type viscometer, obtained values for the viscosity of nitrogen at 25°C., 50°C. and 75°C. and in the range 10 - 1000 atmospheres. The results of these authors are in excellent agreement with other authors at the lower pressures although some divergence exists at high pressures. The result shown for these authors in table 1 is an extrapolated value.

A method of eliminating the end effects found in capillary methods was evolved by Bond[66] in 1936 and applied to measure the viscosity of air. This apparatus was used by Rigden[47] in 1938 for measurements on air, nitrogen and oxygen and a study of his paper suggests an accurate and carefully handled experiment. The accuracy claimed is ± 0.038% and since every precaution appears to have been taken and all corrections have been applied, this may be justified. Measurements were limited to within two degrees of 17°C. so that the value of the temperature dependence of viscosity found by Rigden applied only to this region.

Experiments on a large number of gases, including nitrogen, were carried out by Johnston and McCloskey[68] in the temperature range 100 - 300°K. and at atmospheric pressure. These experiments were carried out on a capillary apparatus and the result given in Table 1 is an interpolated one.

Vasilesco's[69] capillary determinations also show a high degree of experimental accuracy although the apparatus, in both forms, was designed primarily for high temperature work. His measurements were made at atmospheric pressure and at temperatures up to 1600°C. The quoted temperature dependance of viscosity is stated by the author to apply to the temperature range 0 - 280°C.

In 1949 Hirschfelder, Bird and Spotz[70] derived a theoretical equation for the viscosity of nitrogen from the equations given by Chapman and Cowling[71]. The validity of this equation is limited since the assumptions made to formulate it are not fully known.

A correlation similar to the present one was performed by Kestin and Pilarczyk[51] for their oscillating-disc measurements. They concluded that the temperature dependence of viscosity should be taken as 4.55×10^{-7} poise/°C. and recommended $\gamma_{21°C} = 1755 \times 10^{-7}$ poise to an accuracy of 0.2%. This corresponds to $\gamma_{25°C.} = 1773 \times 10^{-7}$ poise. In the re-evaluation of this work by Kestin and Wang[52] the same temperature dependance of viscosity is suggested

but the recommended value of viscosity is $\eta_{25^\circ C}$ = 1778 poise to an accuracy of 0.1%. These two statements are just within the accuracies quoted.

Recently Kestin and Leidenfrost,[59] using an oscillating disc viscometer and the theory of Newell, determined $\eta_{25^\circ C.}$ = 1779 poise. The corresponding temperature dependance of viscosity may be deduced to be 4.7×10^{-7} poise/°C. between 20°C. and 25°C. The absolute accuracy of this determination is claimed to be ± 0.05% and since the experimental work appears to have been carried out with considerable care this may be possible. The design and operation of the instrument used are described in detail in a separate publication[72] which appears to substantiate the claimed accuracy.

The results obtained by the above authors are shown in Table 1. From these results a temperature dependance of viscosity of 4.7×10^{-7} poise/°C. is accepted and this value is used to convert Yen's result to 25°C. The mean value of the experimental results quoted in table 1 gives $\eta_{25^\circ C.}$ = 1776.5 poise to an accuracy of +0.2%, - 0.14%. No result quoted in table 1 is outside this value and it seems reasonable to accept this as the accuracy of the known value of the viscosity of nitrogen.

TABLE I

DYNAMIC VISCOSITY OF NITROGEN

AUTHOR	METHOD	DATE	$\gamma_{25^\circ C} \times 10^7$ poise	Temp.Dep. of Viscosity $\times 10^7$ poise/°C.
1. Yen	Rotating Cylinder	1919	1774	-
2. Michels & Gibson	Capillary	1932	1775	4.84
3. Rigden	Capillary	1938	1776	4.76
4. Johnston & McCloskey	Capillary	1940	1778	4.5
5. Vasilesco	Capillary	1945	1776	3.9
6. Kirschfelder	Theory	1949	1776	4.65
7. Kestin & Pilarczyk	Correlation	1954	1773	4.55
8. Kestin & Wang	Correlation	1957	1778	4.55
9. National Bureau of Standards		1955	1777	
10. Kestin & Leidenfrost	Oscillating Disc	1959	1779	4.7

(b) Air. The first accurate determination of the viscosity of air by a rotating cylinder method was made in 1912 by Gilchrist[73]. These measurements were made at 20.2°C. and an accuracy of 0.2% was claimed. Gilchrist's paper includes a list of eleven previous determinations of the viscosity of air. These have been scrutinised and it appears that the works of Rankine[19] and Rapp[74]

are the only ones worthy of further consideration. Rankine's determination was made at 10.6°C. and Rapp's at 26°C: both used capillary instruments.

The more important subsequent determinations by the rotating cylinder method are those of Houston[75], Kellstrom[76] and Bearden[77]. The most accurate of these is that of Bearden but the accuracy of the other two is sufficient to warrant discussion.

Houston obtained a value at 22°C. and quoted his accuracy as ± 0.25%. All necessary corrections were applied including one for the moment of inertia of the air carried round by the suspended cylinder when its own moment of inertia was being measured and one for the effect of the gap between the suspended and guard cylinders. He did not divide the viscous drag on the gap equally between the guard and suspended cylinders but, because his suspended cylinder was of thin section compared to the guard cylinder, he divided the drag in the ratio 0.47 : 0.53 respectively. This last correction may be slightly in error but the comparatively large uncertainty of the final result must be attributed to experiment and measurement rather than to theory.

The first determination of the viscosity of air by Kellstrom was made at atmospheric pressure in 1937, his subsequent experiments being carried out at pressures up to 30 Kg/cm^2 in 1941. A rotating cylinder, shorter than, but otherwise similar to that

of Houston was used. Experiments were carried out with two outer rotating cylinders of slightly different diameters and a value for viscosity was obtained at 20.0°C. The accuracy of the result is claimed to be ± 1.0 in 1820.1 x 10^{-7} poises, but since no correction was made for the gap between the guard and suspended cylinder this is obviously in error. If this correction is applied the result becomes 1818.1 x 10^{-7} poises and it seems likely that the experimental accuracy of ± 1 remains valid.

The best precision determination is that of Bearden and involves an apparatus in which the outer cylinder is suspended and the inner one rotates. The Reynold's numbers used are so small that no question of turbulence arises. All required corrections were applied and the experiments appear to have been carried out with the utmost care and justify the claimed accuracy of ± 0.003%.

The capillary experiments of Bond[66] and Rigden[67] were mentioned briefly in 2.1(a) above. Both these experiments used essentially the same apparatus for their determinations on air but the result given by Bond is 0.2% higher than that of Rigden. Bond claimed an accuracy of ± 0.8 in his value of 1834.3 x 10^{-7} poise for 23°C and Rigden ± 0.7 in a viscosity value at 23°C. of 1830.0 x 10^{-7} poises. No explanation of this discrepancy was given by Rigden. Several possibilities exist which explain the

error and these suggest that the result given by Rigden is the more reliable. The light paraffin oil used by Bond to establish his pressure difference may have vapourised or his apparent lack of precautions to ensure the constancy of the temperatures along both capillaries may have resulted in error. These possibilities and the fact that Rigden's paper suggests a very accurate and carefully handled experiment have led the author to accept Rigden's value in preference to that of Bond.

Rigden's work is further substantiated by the experiments of Sixsmith[78] and Dolphin[79] using a similar apparatus incorporating two glass capillaries in parallel. Dolphin's method of creating a steady flow through his capillaries is of particular interest and appears to have given excellent stability. The pressure at outlet from his capillaries was lowered to just below atmospheric by means of a pressure balance. This caused a capacity manometer to activate a servomotor which in turn caused a piston to move in a cylinder thereby displacing air at a steady rate. The movement of the piston was recorded by an electronic timer and the rate of displacement was, therefore, known. The accuracy of Sixsmith's work was claimed as ± 0.07% and of Dolphin's, better than ± 0.02%. No published data is available for the former but the latter appears to be justified.

Plattanaik and Banerjea[80] determined the viscosity of air to a claimed accuracy of ± 0.14% by a capillary method and Johnston and McCloskey[68] also obtained a value in the course

of their experiments.

One of the eleven gases measured by Kestin and Leidenfrost[59] was air and they obtained a value of $\eta_{23^\circ C}$ = 1835.5 poise. Again the result is higher than the values obtained by other methods but the claimed accuracy of ± 0.05% appears justified. It remains to explain the descrepancy between this result and the one given by Bearden.

Table 2 lists the experimental determinations which have been described above and table 3 the values recommended by the authors after correlation. The result given in table 3 by Hirschfelder is derived from theory in a similar manner to that obtained for nitrogen in table 1. An average of the values given by sources (1),(2),(3),(4),(7),(8),(9),(10),(11) gives $\eta_{23^\circ C}$ equal to 1831.6×10^{-7} poise to an accuracy of +0.21%, -0.175%. The values given by correlation fall within this accuracy except that of Keyes. The value for the temperature coefficient of viscosity which has been accepted by the author is 5.00×10^{-7} poise/°C. and the assumed accuracy of this figure is ± 5%.

From this investigation it appears that there is little to choose between dry air and nitrogen with regard to the known accuracies of their viscosities. It seems likely, however, that the viscosity of gases may be measured or predicted to even greater accuracies and since nitrogen may be obtained to such a high degree of purity the author agrees with Kestin, that, in principle, nitrogen is the better calibration fluid.

TABLE 2

DYNAMIC VISCOSITY OF AIR

AUTHOR	Method	Date	23°C x 10^7 poise	Temp. Dep. of Viscosity x 10^7 poise/°C
1. Gilchrist	Rotating Cylinder	1912	1828.4	-
2. Houston	"	1937	1829.1	-
3. Kellstrom	"	1937	1833.0	-
4. Bearden	"	1939	1833.79	-
5. Rankine	Capillary	1910	1827	-
6. Rapp	"	1913	1824	-
7. Rigden	"	1938	1830.0	4.93
8. Sixsmith	"	1941	1830.7	-
9. Dolphin	"	1950	1830.8	4.86(20-30)°C 4.89(10-20)°C
10. Plattanaik	"	1938	1833.7	-
11. Kestin & Leidenfrost	Oscillating Disc	1959	1835.5	5.36

TABLE 3.

DYNAMIC VISCOSITY OF AIR - RECOMMENDED VALUES

Author	Method	Date	23°C x 10^7 poise	Temp.Dep. of Viscosity x 10^7 poise/°C
Hirschfelder	Theory	1949	1833	4.75
Dolphin	Correlation	1950	1831.5	-
Bonilla	"	1951	1833	-
Keyes	"	1952	1834	-
Kestin & Wang	"	1957	1830	-

The use of nitrogen as a calibrating fluid would have involved some modification to the calibration apparatus and, since results were of primary importance, calibration with nitrogen was temporarily postponed. The additional uncertainty introduced by not calibrating with nitrogen is negligible.

2.2. WATER AND STEAM

The experimental work on the viscosity of water and steam falls into categories which will be discussed separately.

(a) Experiments at atmospheric pressure and below.

(b) Experiments at pressures above atmospheric.

The experiments in section (b) are described in some detail since the apparatus used for these experiments are in some respects suitable for the conditions of pressure and temperature investigated by the author.

(a) The theory of Maxwell[1] was applied by Kundt and Warburg[81] and Puluj[82] to the measurement of the viscosity of several gases by an oscillating disc method. Virtually the same instrument was used by both authors and each obtained one experimental point for steam. Kestin[40] has estimated the accuracy of these works as not better than 3% and 2% respectively, his estimation being based on a comparison of his own results for air and hydrogen with those obtained by Kundt and Warburg and Puluj.

This estimation appears to be optimistic since Maxwell's theory may have introduced errors up to 5% and a further error

of unknown magnitude may have been introduced by the use of bifilar suspensions causing slight axial movement of the disc to be observed. The results of these authors are, therefore, not included in the correlation of results.

Further experiments with the oscillating disc, and using Maxwell's theory, were carried out by Vogel[83] and later by Braune and Linke[84]. They were, however, relative measurements and Maxwell's theorem when applied to relative measurements introduces an error of the order of 0.5%. Vogel's measurements were related to air at 0°C. and his calibration error was not greater than 0.5%. This experimental accuracy was high but his measurements are restricted to 29°C.

Braune and Linke with a similar accuracy made observations over the temperature range 20 to 400°C. calibrating their apparatus with air at 17°C. The accuracy of the results obtained by Vogel and Braune and Linke may be considered as better than 2%.

These four experimenters obtained their results at pressures below atmospheric in order to have the fluid in the gaseous state. Pressures ranging from 14 mm. of mercury to 210 mm. of mercury were used but pressure was found to have no measureable effect.

C.J. Smith[85] used an apparatus based on Rankine's method of measuring the viscosity of a volatile liquid. This method depends on the transpiration of a superheated vapour through a capillary. He obtained results at 100, 150, 200, 212 and 260°C.,

the pressure again being well below atmospheric. He did not, however, correct for the effect of flow at the entrance to and exit from the capillary and his results are high by amounts up to 0.5%. The experimental accuracy appears to be of the order of ± 1% giving a total accuracy of not less than ± 2%.

A valuable contribution to this field of work was made in 1952 by Swindells, Coe and Godfrey[86] at the National Bureau of Standards. These authors measured the dynamic viscosity of water at atmospheric pressure and at a temperature of 20°C by means of a carefully constructed capillary circuit. Using four different capillaries they obtained $\eta_{20°C}$ = 0.010019 poise and quoted an accuracy of ± 0.05%. This work appears to be of the highest standard and no fault can be found with the experiment, corrections applied or evaluation of viscosity. The accuracy of experiment is indicated by the measurements of the corrections made by the authors.

Although this experiment was carried out with great care the final result must be held in doubt. In the course of his measurements with the oscillating sphere, Moszyński[39] measured the viscosity of three samples of water before and after compression with the results shown in table 4.

The results of these experiments appear to indicate

TABLE 4

VISCOSITY OF DECOMPRESSED WATER

Sample 1		Sample 2		Sample 3	
Press. atm.	20°C centipoise	Press. atm.	20°C centipoise	Press. Atm.	20°C centipoise
1	1.0051	1	0.9840	1	-
340	-	341	0.9768	340	+
3.73	0.9894	1	0.9895	1	0.9877

that the viscosity of water is stable only after compression or that the viscosity of water may change after compression. These results are not conclusive, however, as only three samples were used and since the second sample was reported to be 'stale distilled water'. Further work is being carried out at Brown University to attempt to verify this phenomena and the indication appears to be that the viscosity of water has one value prior to compression and one value subsequent to the compression.

The viscosity of steam at atmospheric pressure and high temperatures has been measured recently both in the U.S.A. and U.S.S.R. The results of the former were presented in two papers by Bonilla, Wang and Weiner[87] and Bonilla, Brooks and Walker[88] and gave experimental results in the temperature range 450 - 1200°C. These results were consistent and fitted

their smoothed curve to ± 0.5%. Little experimental data is described and a critical analysis is not possible. Their results for argon and nitrogen agree closely with those obtained by Vasilesco[69] and this is in their favour since the work of Vasilesco is of a high accuracy. A coiled capillary was used and White's[89] correction for coiling applied. Corrections for end effects and slipping were applied.

The thermocouples used by these authors were platinum 10% rhodium-platinum and the average deviation of temperature amounted to about 4 C^{o}. If these thermocouples were not calibrated - and there is no mention of calibration - then the accuracy of the thermocouples cannot be guaranteed to better than ± 1 C^{o} at temperatures up to 1100°C. causing a total average inaccuracy of ± 5 C^{o}.

The determinations made in the U.S.S.R. were by Shifrin[90] and are in the temperature range 149 - 866°C. A two capillary method is used with pressure tappings before, between and after the capillaries. An open circuit system was used and the entire mercury manometer and connecting tubes were maintained at a temperature above the boiling point of the fluid. The experiments were carried out using both glass and quartz capillaries and an accuracy of between 1.5 and 2% was claimed by the author. The computed values do not deviate from the

smooth curve by more than ± 1%.

The results of Shifrin differ from those of Bonilla by a constant amount of 3.8% and it appears that a systematic error must exist in the work of one or other of these authors. Shifrin's apparatus appears the less likely to have a systematic error while Bonilla's use of a coiled capillary may have introduced such an error.

(b) The first investigation of the pressure dependence of viscosity was made by Speyerer[91] in 1925. His apparatus was an open circuit capillary type viscometer, the brass capillary being 240 cm. long and 0.19 cm. inside diameter. The capillary was divided into three sections joined together by means of sleeves. Each of the two sleeves was provided with a slot which served as a pressure tapping, see Figure 1. Speyerer assumed that these slots would have no effect on the flow and that by measuring the pressure drop across the centre portion of the capillary - 200 cm. long - he would eliminate end effects. The accuracy claimed by Speyerer was 4% but his results show a scatter of up to 10% from the mean and do not appear reliable. Measurements were made in the pressure range 1 to 10 atmospheres and at temperatures from 100 - 350°C. The inaccuracies are probably due to fluctuations in pressure and flow, errors in pressure measurement and possibly to bad location of the steam-water interface.

Sigwart attempted to criticise Speyerer's work on the grounds of end correction and he applied a correction to allow for same. Although Sigwart's correction is too large he is correct in stating that Speyerer over-estimated the pressure dependence of viscosity because of his pressure tappings. It seems likely, however, that the effect of these tappings on the flow would be small and, compared to the experimental accuracy, negligible.

In 1934 Schiller[92] published the results of his experiments in the range 1 - 30 atmospheres and 100 - 300°C. The experiments were carried out by observing the variation in the coefficient of discharge of a nozzle - the nozzle having previously been calibrated with water. The pressure dependence of the viscosity of steam was found to be very large. This method of determining viscosity depends on the assumption that the coefficient of discharge is a function only of the Reynolds number. The range of Reynolds number used by Schiller was 9×10^3 to 70×10^3 which with his configuration implies turbulent flows. Since the flow is turbulent throughout his experiments, the flow for calibration purposes will only be approximately equal to that for the measurements on steam. A graph of coefficient of discharge against Reynolds number was drawn up for the water calibration but conditions for steam were unlikely to be similar.

Schiller's results are, therefore, considered of little value.

Schugaiev[93] used a Rankine type viscometer to measure the viscosity of steam in the range 1 - 93 atmospheres and 100 - 400°C. The capillary was 27 cm. long and was calibrated with air but no correction was applied for compressibility or slip. Mercury was used as the driving fluid at the lowest temperatures and tin at all other temperatures. It is possible that the use of tin may have caused the high scatter experienced at the high temperature. The accuracy claimed by the author was ± 1% at 100 C°, ± 2.5% at 250 C°, ± 5% at 350 C° and ± 6.5% at 400 C°. Schugaiev, himself, concluded that the pressure dependence of viscosity could not be determined from his experiments because of the large scatter in his results. The subsequent report by Schugaiev and Sorokin[94] containing further information from the same apparatus must also be considered unreliable at the higher temperatures due to the large scatter experienced.

The results of Sigwart[95] were obtained from an open circuit viscometer similar to that used by Speyerer. Two capillaries were used in the experiments, one of platinum with a bore diameter of 0.0547 cm. and a second of quartz with a bore of 0.0375 cm. Both capillaries were 36 cm. long and end corrections were applied. The measurement of pressure drop across the capillary was effected by means of

a ring balance. It was found, however, that slight movements of this balance caused water to flow through the connecting tubes and into the capillary. For this reason Sigwart was forced to calibrate his balance and this he did by carrying out two experiments in which the ring balance was in different positions and two different flows were obtained. The range of temperatures was 275 - 380 C^{o} and of pressures 25 - 270 atmospheres and Sigwart estimated his error to be a maximum of ± 3% at the maximum temperature attained. The experiments appear to have been carried out with great care but the apparatus is subject to the usual difficulties of an open circuit viscometer - stability of flow and pressure measurement. Also it appears that manometer readings were taken before the flow was entirely stable and this may have been the cuase of the scatter experienced at the higher temperatures. Sigwart's attempt to extrapolate his results to 500°C. is incorrect due to his assumption that the viscosity of steam and the viscosity of water behave in the same manner with respect to temperature.

Three separate determinations of the viscosity of steam have been made at Purdue University. The first in 1935 was by Hawkins, Solberg and Potter[31] who used a falling ball viscometer and applied the theory of Lawaczeck[96]. The results of this experiment were superseded by those obtained

by the same authors in 1940 using a capillary viscometer[18]. The error in the 1935 results was attributed to a high Reynolds number and to incorrect assumptions made in the development of the theory. No attempt to correct the results was made by the authors. In a contribution to the 1935 paper, Schugaiev[97] attempted to recalculate the results by introducing an "end correction". This approach does not appear to have sufficient justification.

The results published in the 1940 paper covered the range 5 - 120 atmospheres and 180 - 550°C. The capillary used was 103.2 feet long and 0.09294 inches inside diameter. These dimensions were said to be justified by the elimination of end effects and the measurability of the pressure drop. Steam was supplied to the capillary from a boiler and was condensed and weighed in the usual way. Pressure measurement was effected by a mercury in metal manometer. The apparatus was calibrated for Reynolds numbers above 180, water and nitrogen being used as the calibrating fluids. The results obtained do not appear to be reliable and a scatter of ± 10% was experienced at the highest pressures. This error is probably due to the effect of pressure on the coiled capillary and the consequent alteration in bore dimension. The absence of a thermal expansion correction and the difficulty in obtaining stable conditions of temperature and pressure and Reynolds number would also introduce errors.

The apparatus used by Savino and Sibbitt[98] for the measurement of the viscosity of nitrogen and carbon dioxide was used for measurements on the viscosity of steam by H.H. Osborn[99][100]. This apparatus consisted of an open circuit capillary viscometer, the capillary being 500 feet long and 0.082 inches inside diameter, and it was designed for a maximum pressure of 500 atmospheres and a maximum temperature of 650°C. This capillary was coiled into a 10 foot diameter and located in a horizontal, annular, electrically heated furnace. Savino and Sibbitt calibrated the apparatus with nitrogen and found that for values of Reynolds numbers greater than 250, the calibration constant depended upon the value of the Reynolds number, i.e. the flow was not laminar. The results for nitrogen and carbon dioxide showed a scatter of ± 3% at low and medium pressures. At the highest pressures investigated, 280 atmospheres for nitrogen and 50 atmospheres for carbon dioxide, a scatter of ± 4% and ± 6% respectively, was found. An error of ± 2.5% was estimated for calibration. The application of White's coiling correction may have introduced a further systematic error. These authors concluded that their apparatus was a reliable and accurate method of measuring the viscosity of inert gases and other single phase fluids at high temperatures and pressures. It appears that on this recommendation Osborn undertook his work on steam. The conclusions reached by

Savino and Sibbitt appear to be optimistic since an apparatus giving a minimum random error of ± 4% can hardly be considered reliable or accurate. Nevertheless Osborn has observed values of the viscosity of steam at 450, 500 and 900°F. and at pressures up to 1000 p.s.i.a.

On the basis of his results, Osborn concluded that the rapid increase in dynamic viscosity indicated by Timrot[101] neat the saturation line might be in error and the dependence of the viscosity on pressure is not as great as that given by Timrot. The first conclusion must be considered doubtful since an apparatus of the capillary type is not ideal for measurements in this region. The second conclusion, however, may be justified since the results indicate a deviation from Timrot's which cannot be accounted for by the stated uncertainties.

The potential inaccuracy and scatter in results obtained by coiled capillaries, such as those used at Purdue University, has led the author to believe that this form of apparatus is not ideal for the accurate determination of the viscosity of steam.

Results in the range of pressure 93 - 352 Kg/cm.^{2} and of temperature 19 - 601°C were published by D.L. Timrot[101] in 1940. An accuracy of 2% was claimed at temperatures up to 250°C and 3% at higher temperatures. A Modified Rankine viscometer was used incorporating a platinum capillary 38.45 cm. long and

0.04224 cm. inside diameter. The pressure difference across the capillary was obtained by means of a ring balance containing mercury. Displacement of the ring and the mercury contained in it caused the mercury to gravitate to its original position driving water before it and causing steam to flow through the capillary. The apparatus was suspended on knife edges with the capillary in the horizontal position. Corrections were applied for thermal expansion, effect of the ends and pressure drop in the connecting tubes. No fault could be found with his evaluation or with any of the corrections applied. The bore diameter of the capillary was quoted to a considerable accuracy but no details of the method of obtaining this dimension were given.

In general, Timrot's results agree with his smoothed curve to within 2% but at 200 Kg/cm^2 and 400 C^o a discrepancy of 5% exists, at 150 Kg/cm^2 and 500 C^o 4%, and at 304.5 Kg/cm^2 and 446 C^o 4.7%. The results of Timrot were used by Vukalovitch[102] as the basis for his viscosity tables. These smoothed curves also show some deviations from Timrot's experimental results, notably one of more than 5% at 300 Kg/cm^2 and 440°C. The only general trend to be found in these results is that the experimental values of Timrot are generally higher than those given by the tables of both Vukalovitch and Timrot, themselves. Little adverse criticism of the work described in Timrot's paper can be made and his claim of 3% accuracy appears possible

unless a systematic error exists. The stated deviation of certain experimental points from the smooth curve may have been due to the difficulty of operation of the viscometer.

Chromel-alumel thermocouples were used in the viscometer and were carefully calibrated. No mention was made of re-calibration. It is known[103] that chromel-alumel thermocouples are not stable in an oxidising atmosphere such as that used by Timrot and if they were not recalibrated then it is probable that his temperatures are in error by up to 2 C^{o} depending upon the length of time in use at the higher temperatures, the gauge and quality of the wire. The specimen calculation given by Timrot shows a temperature gradient across the capillary of $5^{o}C$. If this is a normal temperature gradient throughout the experiments then the temperature accuracy may be of the order of $\pm$ 4 C^{o}.

It appears that Timrot's results are the most reliable found in the literature to date. The low pressure results of Timrot, Hawkins and Sigwart are similar but at higher pressures considerable divergence is experienced.

An apparatus of the open circuit type but using an annulus in place of the normal capillary was used by T.W. Jackson[104] to measure the viscosity of steam in the range 1 to 100 atmospheres and 100 - 650 C^{o}. It was hoped that the annulus would provide a measurable pressure drop and flow while maintaining an apparatus of suitable dimensions to facilitate

good temperature control. It was reported, however, that the apparatus was operating under turbulent conditions for Reynolds numbers greater than 265 and that a reliable dimension for the inside diameter of the outer tube could not be obtained. The apparatus was, therefore, calibrated for all pressures and temperatures and values for the viscosity of steam were obtained. The possible error in these results is of the order of 7%.

In an attempt to improve the accuracy and range of the annulus viscometer Jackson and Thomas[105] reported a modified apparatus in which the limitations of the 1949 apparatus were improved. The boiler reservoir was eliminated since it could not be used at or above the critical pressure and a variable stroke pump, surge chamber, heated tank and high pressure drop nozzle were substituted. The pump was designed for 2000 atmospheres and it was hoped that pulsations would be eliminated by the surge tank. The new design also included an electric probe manometer to replace the X-ray measurements at pressures above 300 atmospheres. For lower pressures it was hoped to use a visual system.

The results of experiments carried out on this apparatus were published by Jackson and Thomas[106] and in greater detail in the final project report[107]. They cover a range of pressures from 67 p.s.i.a. to 4525 p.s.i.a. and a temperature

range from 434°F. to 881°F. No accuracy is quoted for these determinations but the authors conclude that the best available results for the viscosity of the saturated vapour are those of Sigwart and that the data given by the Russian Steam Tables indicate a variation of viscosity with pressure which is too high. An equation is given for the viscosity of steam at temperatures up to 500°C. and for densities below 0.04 gm/cm.^{2} In this region the average deviation of the experimental data is 1.88% and the maximum deviation 6%. A second equation is suggested for higher densities but no information regarding its validity is given. The results indicate that no significant variation in viscosity could be detected for pressures up to 100 atmospheres.

The most recent measurements of viscosity are those of E. Kjelland-Fosterud[60][61] who used essentially the same apparatus as used later by the author. Measurements were made, relative to the viscosity of air, at pressures ranging from 200 - 1000 Kg/cm.^{2} and at 430 C°. At 380 C° and 540 C° measurements were also made but since no calibration constant was obtained for the drop tube and capillary no true values of viscosity could be obtained. The gradient of constant pressure lines on the graph of viscosity against temperature were approximately the same as those found by Timrot. Further observations were made at 540 C°, 680 C° and 750 C° but the results had to be neglected due to the accidental partial

blocking of the capillary with gold solder. The accuracy of the results obtained at 430 C^{o} was believed to be ± 2.5%.

The values obtained at 430 C^{o} agree with those of Timrot to within the accuracies of the two experimentors. Subsequent experiments by the author have shown that the temperatures and pressures quoted by Kjelland-Fosterud are in error by amounts shown in the appendices A and B.

The last viscosity results to be discussed are those obtained by Moszynski[39] using the apparatus described by Kestin and Moszynski[38]. This apparatus was used for measurements on compressed water - with an oscillating sphere - and for measurements on steam - with a disc oscillating between two plates. The former results are those presented by Kestin[40] at the International Conference on the Properties of Steam in 1958 and partly discussed in 2.2(a). It is of interest that the results of Timrot are in general higher than those of Moszynski and in particular one point at 203°C. and 350 atmospheres is some 17% high. The Moszynski results agree well with those of Sigwart.

The steam results are in the temperature range 137 - 267°C and in the pressure range 1 - 42 atmospheres and the absolute accuracy is claimed to be ± 1% although the relative accuracy is believed to be considerably better. The results indicated a decrease of viscosity with increasing pressure, this decrease becoming progressively smaller as the temperature rises. At

the highest measured temperature the viscosity was found to increase again for pressures above 35 atmospheres.

The above review of the literature relevant to the measurement of the viscosity of water and steam indicates that the value of the viscosity is still subject to considerable doubt at high pressures and temperatures. The range of pressures and temperatures investigated by the author has not been previously attempted with the exception of the preliminary results obtained by Kjelland-Fosterud. Further experimental results are required at most conditions and independent checks are particularly necessary in the range of conditions covered by Kestin and Moszynski, Timrot, Bonilla and Shifrin.

CHAPTER 3

DESCRIPTION OF APPARATUS.

The apparatus is essentially that used by Kjelland-Fosterud for his determinations, but has been modified as detailed below. It is a closed circuit, capillary type, viscometer which operates at constant pressure and combines Rankine's method of displacing the fluid with Timrot's use of concentric flows, through the capillary and back through the annulus formed by the capillary holder and the pressure tube. A schematic flow diagram is given in figure 2.

A mercury pellet of known mass falls, due to gravity, in the 2 mm. bore pyrex drop tube and displaces the water which fills the apparatus. The water passes into the connecting tube, which holds the capillary, and is converted into steam before passing through the capillary at conditions of constant temperature and pressure. The rate of flow is found by timing the fall of the mercury pellet in the glass tube over a known length.

The bore of the capillary, and the volume corresponding to the timed length cannot be measured accurately by a direct method. Hence the drop tube and capillary are calibrated in a separate apparatus using clean, dry air as the calibrating medium. This apparatus is shown in figure 3 with the drop tube and capillary in position. Water, whose temperature is maintained constant by means of a toluene regulator and 250 watt heater, is

circulated through the annulus surrounding the chamber containing the drop tube and capillary and maintains the air at a steady temperature. The calibration apparatus is designed to allow rotation about its centre and so permit calibration for both directions of flow. In the same way the pressure tubes of the viscometer are capable of 180° rotation to allow measurement with the mercury pellet falling in either direction. Journal bearings are provided outside the furnace to permit this.

The pressure vessel consists of two Numonic 80A tubes 9/16" outside diameter, 5/16" bore and 900 mm. long. These are connected to a central body of 18/8 stainless steel. All pressure joints are effected by means of lens rings. Nimonic 75 is used for the connecting tube which is silver soldered to a 90° bend at the cold end and joined to the capillary at the high temperature end. The previous use of an 18/8 stainless steel connecting tube and capillary has been discontinued due to unsatisfactory corrosion resistance at the higher temperatures.

The furnace is made up of four heaters - one main heater capable of supplying 1½ kilowatts and controlled by means of fixed resistors which can be arranged in series or in parallel, two disc heaters to overcome end losses, and a tube heater to reduce the heat lost by conduction along the pressure tube. The disc heaters have a possible dissipation of 350 watts and the

tube heater of 400 watts. Control of these heaters is effected by means of fixed resistors and slide wires which give an infinitely variable power dissipation within the capacity of each heater. The a.c. mains supply is stabilised by means of a constant voltage transformer and converted to 110 Volts.

The pressure in the viscometer is raised by means of an oil pump which necessitates an oil-water interface. To avoid possible contamination of the water a rubber diaphragm is located and held in position by means of a specially recessed lens ring. The comparative incompressibility of the water and the small volume of compressible steam ensures that the displacement of the diaphragm is small enough to allow the design pressure of 1000 $Kg/cm.^2$ without straining the diaphragm itself.

The features of the apparatus which have been modified by the author are now considered in greater detail.

3.1 CAPILLARY

The capillaries previously used in the apparatus were of 18/8 stainless steel and platinum/10% rhodium alloy. The former was eventually discarded for temperatures above 500°C due to corrosion, and considerable difficulty was experienced in sealing the joint between the latter and the connecting tube.

The seal used for the stainless steel capillaries is shown in figure 4a., the sealing face being silver soldered to the capillary. This seal proved effective and was successfully detached after use at 540°C. For measurements above 500°C it was considered desirable to use the platinum alloy capillaries and these were gold soldered to the connecting tube as shown in figure 4b. Lack of experience in the technique of gold soldering resulted in the capillary being partially blocked with gold solder. This was not discovered until results had been obtained at 680°C and 750°C. These results were some 12% lower than the extrapolated values of Timrot and this suggested the checking of the calibration constant. Unfortunately the capillary sheared when the gold soldered joint was taken apart. The remaining portion of the capillary was then calibrated and when allowance was made for the missing length, the capillary constant proved to be within 0.5% of the original value. A specimen of the blocked portion of the capillary was mounted and after etching and microscopic inspection it was concluded that gold solder had entered the capillary. This can only have happened when the joint was originally soldered.

The remaining section of the capillary was cut into three parts and these specimens were mounted, etched and polished. Microscopic inspection revealed that the bore was not exactly round and that intercrystalline cracks appeared to have formed

in the radial direction. This may have been due to the steam itself, to impurities in the steam or to the gold present in the capillary. Dynamic tests in this laboratory indicate that the presence of high purity steam at atmospheric pressure and at 800°C does not cause intercrystalline cracking in platinum/10% rhodium alloy. This suggests that the cracking is a result of the presence of gold and not of the steam or its impurities. This, however, is not certain and indicates the need for calibration of the capillary before and after use at high temperature.

The capillary must, therefore, be removed from the connecting tube after each isotherm and a gold soldered joint does not permit this. Experiments were carried out on alternative types of seal and the most successful proved to be a screwed adapter threaded 2 B.A., sealing on a 20/22° cone joint as shown in figure 4c. Some difficulty was experienced in the manufacture of this member, but trial and error showed the best method to be a machined face, lapped with a brass former.

A platinum/10% rhodium alloy capillary was gold soldered to the adapter by Messrs. Johnson, Matthey. This capillary was 300 mm. long x 0.20 mm. nominal bore.

The cone angle seal is tested before the apparatus is assembled by passing air at 30 lb/in.^{2} through the capillary at room temperature and observing the seal under water. The pressure difference to which the seal is subjected in the apparatus is of the order of 2 cm. of mercury. To verify that no leak is present

when the seal is inside the apparatus and subjected to working temperatures the apparatus is taken apart after a number of determinations of viscosity have been made. The seal is then rechecked using compressed air, unscrewed, retightened and again tested under air pressure. The apparatus is then reassembled and the previous viscosity determination repeated. Provided the results obtained in each case are within 1% of each other it is assumed that the seal was tight in the first instance and is again such that all the steam passes through the capillary.

The above precautions have been successful and no difficulties were experienced in dismantling the joint at any temperature. No leaks were detected during any of these measurements. The calibration constants agreed well before and after all isotherms except that at 540°C. where the calibration proved to be 5% lower than the original calibration. The calibration was checked for blockage by passing a 0.0065 in. wire through it and no obstruction was found. The capillary and drop tube were carefully recalibrated and this calibration constant used for the determination of the viscosity of steam at two pressures. These tests indicated that the results obtained using the previous calibration constant were in order. The change in capillary bore must, therefore, have taken place after the measurement of viscosity at 540°C. and before the recalibration. This suggests that a local blockage, due to the capillary being slightly deformed on removal, is the most likely reason for the change in

capillary constant. The new value of the constant was checked after the second measurement of viscosity at the two pressures and was found to be unchanged.

A new capillary was procured for all subsequent measurements.

3.2 DROP TUBE

Measurements of viscosity on the 480°C. isotherm were made using a drop tube, previously used by Kjelland-Fosterud, and calibrated with a new capillary. This drop tube had four timed lengths in each direction of flow and had, therefore, sixteen 0.008" platinum contact wires inserted through its wall.

The disadvantages of this arrangement are threefold -

(a) A wire cannot be sealed into the wall of the pyrex glass tube without causing some irregularity in the bore of the tube. It is therefore, advantageous to minimise the number of contact wires and possible irregularities.

(b) A large number of timed lengths, implies shorter timed lengths and, therefore, shorter timings. The stop watch method of timing previously used and used in part by the author is less accurate for short timings and should not, therefore, be used with a short length drop tube, in particular at 380°C and 430°C where the kinematic voscosity is at a minimum.

(c) A large number of contacts require considerable maintenance and cleaning.

The drop tube used to measure the viscosity at the 380°C and 430°C isotherms and also for pressures above 400 Kg/cm.^{2} on the 540°C isotherm has one timed length in each direction and six wire inserts. This drop tube is shown in figure 5. The timed lengths are nominally 260 mm. and the mean cross-sectional area is approximately 0.03 cm.^{2} A similar drop tube with timed lengths of 100 mm. has been constructed for use at higher temperatures and at the lower pressures on the 540°C isotherm.

The insulation of the live contact wires has been effected in two ways. The first is the use of molten polythene which is allowed to solidify around the wire. This is an excellent insulation but is difficult to remove should the contact wire need re-soldering to the P.V.C. covered 'live' wire. The second method is to surround the joint with surgical rubber tubing. This is easily removed after short periods of immersion in water but deteriorates rapidly and is difficult to remove after longer periods in the water. Rubber insulation was used on the drop tubes at 380°C, 430°C and 480°C. The rubber was then removed and the joint re-soldered as required and polythene insulation substituted.

3.3 TEMPERATURE MEASUREMENT

The temperature in the calibration apparatus is measured by means of a total immersion mercury-in-glass thermometer graduated to 0.1 C°.

The temperature of the steam in the capillary and connecting tube is deduced from five platinum 13% rhodium-platinum thermocouples placed as shown in figure 6. These thermocouples have been in the apparatus since the original construction and are inside open ended stainless steel tubes which carry the wires, suitably insulated, to a terminal block, approximately 12 in. from the end of the furnace. Compensating leads are used to connect the terminals to the cold junction and copper leads from the cold junction to the potentiometer. A sixth thermocouple of similar metals, with compensating leads was used as a probe thermocouple and acted as a check on the other five.

The cold junction is common to all thermocouples and is held at room temperature. The cold junctions of the six thermocouples are sealed in wax within a thermos flask. The temperature of the cold junction is determined by means of a mercury-in-glass thermometer.

The measurement of thermal e.m.f. was originally made on a Pye precision potentiometer graduated to 1 microvolt. Measurements on the 380°C, 430°C, 540°C and 650°C isotherms have, however, been made with a Cambridge potentiometer graduated to 0.1 microvolt.

On the commencement of the work described in this thesis the thermocouples had been inside the apparatus for three years and no check calibrations had been carried out. The accuracy of thermocouples using wire supplied by Messrs. Johnson,

Matthey is quoted by these suppliers as subject to deviations of not greater than ± 1 C° up to the gold point. It was, however, possible that due to the long periods of use at temperatures up to 780°C the thermocouples had become contaminated and were no longer accurate to these limits.

It was, therefore, decided to calibrate the thermocouples in situ in the following manner, since the original thermocouple assemblies could not be easily removed from the apparatus.

(i) The probe thermocouple should be checked against a N.P.L. standard thermocouple.

(ii) A new platinum 10% rhodium platinum thermocouple should be made up and carefully calibrated against the N.P.L. standard thermocouple.

(iii) This new thermocouple - thermocouple C - should then be used to calibrate the existing thermocouples in the viscometer. Since results were the primary consideration, it was decided to limit the calibrations to the thermocouples which give the steam temperature along the length of the capillary, i.e. 2, 3 and 4.

This procedure was carried out and the results are recorded in Appendix A.

Calibration of Thermocouple C.

The accuracy of the N.P.L. standard thermocouple is quoted by N.P.L. as ± 0.1 C° at temperatures up to 600°C and ± 0.3 C° at temperatures above 600°C. The test thermocouple C was made up

from 30 gauge platinum 10% rhodium platinum wire, the hot junction being carefully constructed in the manner recommended by N.P.L. and annealed in a furnace at 1000°C. The thermocouple was sheathed by twin core insulators and surrounded by a stainless steel tube having one end sealed.

The calibration was carried out, in a furnace designed for this purpose, at seven points up to 770°C. and a graph of the results drawn up. This calibration is shown in Appendix A. The interpolation error is believed to be not greater than ± 0.1 C°. The total accuracy of thermocouple C with respect to the International Scale is, therefore, ± 0.3 C° up to 600°C and ± 0.5 C° from 600 - 700°C.

Calibration of Existing Thermocouples.

Thermocouple C was inserted into the pressure tube of the viscometer and placed so that the hot junction of the thermocouple C and of the thermocouple under test, lay in the same radial plane. It is assumed that inside the furnace tube the temperature at the hot junction of the existing thermocouples - all shielded from radiation - should be the same as that at the hot junctions of thermocouple C when in the same radial plane. This will be true only if the thermocouples are in thermal equilibrium with their immediate surroundings. This was assumed to be true and the observations were accepted if thermocouple C could be moved axially ½" in either direction without any measurable difference in e.m.f. This check was carried out before

the calibration of each thermocouple and was found to be satisfied, provided the temperature gradient along the pressure tube was not greater than ± 2 C^{o} between thermocouples 1 and 5. For each test the open end of the pressure tube was filled with a Sindanyo disc designed to reduce axial heat flow. This was found to make no measurable difference to the results.

The radial temperature gradient inside and outside the pressure tube was checked using thermocouple C - a radiation shield being provided when outside the pressure tube. This test was carried out only at 540°C and no measurable gradient could be detected.

The cold junction of thermocouple C was maintained at 0°C and the platinum 10% rhodium platinum wires were taken back to the cold junction. A reversing switch was included in the test circuit to ensure that stray e.m.f.'s were eliminated.

Calibration was carried out before and after the measurement of viscosity at each temperature.

3.4 PRESSURE MEASUREMENT

A diagrammatic sketch of the apparatus previously used to raise, measure and maintain pressure in the apparatus is shown in figure 7. Inspection of the piston used in the dead weight gauge of this apparatus showed it to be diametrically undersize and scored in parts. This had resulted in considerable leakage of oil at the higher pressure and continual make up had been found necessary. To overcome this a new Budenberg type

dead weight gauge was incorporated in the apparatus as shown in figure 8. The original dead weight gauge was calibrated against the new Budenberg gauge. The mass of all weights was checked, and the calibration results are shown in Appendix B.

The piston area of the original dead weight gauge was nominally 0.01 $in.^2$ as compared with the 0.02 $in.^2$ of the new gauge. The latter is based on a piston diameter guaranteed by N.P.L. to 5 parts in 100,000. The weights used on the gauge tester are annular and are guaranteed to within 2 grains of the nominal weight.

It is known that free piston gauges are subject to certain errors. The measurements are based on the following assumptions,

(a) the piston is truly cylindrical and moves in the cylinder with no friction,

(b) the piston and cylinder do not distort under pressure,

(c) there is no leakage of oil past the piston.

The calibration of the piston diameter is sufficiently accurate to allow out of roundness to be neglected. Friction is present in the piston cylinder assembly but is minimised by rotation of the weight carrier and hence the piston. The assembly is rotated for the duration of each time length.

The work of Bett, Hayes and Newitt[108], Johnson and Newall[109], Dadson[110] and Johnson, Cross, Hill and Bowman[114] at N.P.L., indicate that up to the maximum pressure used in the

present apparatus any distortion error may be neglected. Leakage of oil past the piston is also a function of pressure and causes an effective increase in the area of the piston. It has been shown that provided there is no dimensional change, the effective area may be taken as the mean of the cross-sectional areas of the piston and the cylinder.

The makers of the dead weight gauge state that the cylinder is manufactured to fit the piston with a minimum of clearance and it is assumed that any correction required from this source will be less than 0.1% and, therefore, negligible for the purposes of this investigation. The inherent errors of the dead weight gauge will not, therefore, significantly affect the measurement of pressure in the present apparatus.

The continual make up of oil in the original apparatus may have resulted in errors due to pressure fluctuations since the effect of such fluctuations is to drive the mercury pellet and hence the steam, to or from the capillary. Further the length of pressure tubing between the piston cylinder assembly and the viscometer was found to be excessive, since this would reduce the response of the pressure measuring system to possible fluctuations. It is thought that these faults have been overcome by the introduction of the new dead weight piston gauge with suitably shortened connecting pressure tube and the removal of the entire assembly previously used.

With two exceptions all joints and seals in the pressure

vessel and associated pressure-raising equipment are entirely without leakage and are effected by lens rings and cone joints. The first exception is the piston cylinder assembly which has a negligible leakage and whose effective stroke volume more than balances any leakage, even at the highest pressure. The second exception is the rotating seal on the pressure line between the dead weight gauge and the viscometer. This rotating seal is necessary to allow the fall of the mercury pellet to be reversed and causes an alteration in pressure equivalent to the gain or loss in volume corresponding to the axial movement of the thread by one half turn. There is no visible leakage from this rotating joint. A slight increase or decrease in pressure has to be made to counteract this effect but the introduction of the present type of drop tube in which the pellet falls half the length of the drop tube before the timed length commences allows more than enough time for the pressure to stabilise. A timing is not accepted if in the course of it any alteration in the position of the piston of the dead weight gauge is observed.

3.5 TIME MEASUREMENT

The measurement of time was previously carried out by means of a solenoid-activated stop watch and relay incorporated in the circuit shown in figure 9a. No check had been made on the relay delay times and on the consistency of the time lag of of the solenoid. The stop watch was therefore calibrated against a Transisterised Frequency Measuring Instrument manufactured by

Venner Electronics Ltd. (Type TSA2) and capable of measuring time to ± 0.0001 second. The circuits shown in figure 9 were combined for this calibration.

This test showed that no reliable readings could be taken from the stop watch for timings of less than 10 seconds, i.e. one revolution. This was caused by the inability to read the watch with sufficient accuracy since it is graduated in units of 0.025 second. Also, the original N.P.L. certificate states that the error of the watch in any interval of time during the first revolution of the indicating hand did not exceed 0.05 second. This guarantees the watch to 0.5% at best, for this time interval.

The certificate quoted a maximum error of 0.35 second for the longest tested internal of 300 seconds, i.e. 0.12%. The longest interval tested by the author was 200 seconds and the majority of readings were taken for intervals of time from 20 - 70 seconds. The stop watch was found to be consistently slow by amounts ranging from 0.15% to 0.25% when in the 'pendant up' position. This error was found not to depend on the length of timing within the range tested. When in the 'dial up' position the error was never greater than 0.15%. No attempt has been made to correct the recorded timings on the basis of these results since the error is small and not entirely constant. All timings for the 380°C, 430°C and 480°C isotherms were made with the watch in the 'pendant up' position and those at 540°C and 650°C with the watch in the 'dial up' position.

These results showed the need for improved accuracy on short time intervals, in particular those less than twenty seconds. Some doubt must be cast upon the calibration of Kjelland-Fosterud where timed lengths of 5 - 10 seconds were used.

(a) Calibration

The circuit shown in figure 9b was used for the calibration of the old drop tube and platinum capillary used at 480°C and proved successful for runs with a single pellet. Some difficulty was experienced when a double pellet was used to determine the surface tension drag correction. For this drop tube the timings were of the order of 7 seconds and the counter had to be cut out when the first part of the pellet passed through the start contacts and before the second part of the pellet passed through. The counter had then to be cut in before the first part of the pellet reached the stop contacts.

With this difficulty in mind the circuit shown in figure 10 was constructed and proved more successful. Some difficulty was experienced with the relays used in this circuit. They are sensitive to position and to shock and several timings were missed for this reason.

The circuit was used for all calibration timings before and after the measurement of viscosity at the 380°C., 430°C and 540°C isotherms. A more recent model (TSA3) of the same counter was incorporated for these and all subsequent measurements.

Prior to the calibration of the new capillary and drop tube used for the measurements at 650°C., one of the relays in the timing circuit ceased to function and since the circuit had given some trouble, a new one was evolved. This circuit is shown in figure 11 and was used for all calibrations of the drop tube and capillary used at 650°C.

(b) Steam measurements.

The circuits developed for the calibration apparatus were applied to the viscometer, itself, but it was found that after a short time the apparent electrical resistance of the water had fallen from its normal value of greater than 20 KΩ to less than 2 KΩ. The circuit does not function when the apparent resistance of the fluid is less than approximately 1800 Ω thus preventing timings from being obtained.

All timings measured on the viscometer are greater than 20 seconds and all those measured using drop tubes other than the one used for the 480°C isotherm are greater than 90 seconds. The error involved in using the stop watch circuit is small and this has continued to be used.

3.6 WATER

The availability of a supply of pure water has enabled the author to use water of a higher purity than was previously used in the apparatus. The new water supply is treated for use in a supercritical steam generator and hence its mineral content and electrical conductivity are particularly low. This water is

obtained by passing distilled water through a bed of mixed resins. It is then boiled to expel dissolved oxygen and admitted to the viscometer by evacuating the viscometer and allowing the boiling water to flow in.

3.7 MODIFIED APPARATUS FOR GASES

In order to provide a check on the values of viscosity obtained for steam from the present apparatus, it was decided to measure the viscosity of a gas at some temperature and pressure where the viscosity of that gas is well known. It was also hoped to prove the suitability of the apparatus for the measurement of the viscosity of gases at high temperatures and pressures.

The gas chosen as the most suitable for these measurements was nitrogen since considerable experimental work has been directed towards the determination of its viscosity. In spite of the large number of experiments only those shown in table 5 are sufficiently accurate to be worthy of consideration and it is noteworthy that no accurate results appear to be available in the pressure-temperature fields for temperatures higher than 75°C and pressures higher than 70 atmospheres.

To permit these measurements it was necessary to modify the apparatus to make it suitable for use with a gas. The dead weight gauge tester shown in figure 8, was removed, as was the rubber diaphragm which had previously been used to separate the oil and water, and the system shown in figure 12 was substituted. This provides three outlets from the pressure vessel. To one of

TABLE 5

EXPERIMENTAL DATA SOURCES FOR NITROGEN

Author	Reference	Temp. Range °C.	Pressure Range Atmospheres
Kestin & Leidenfrost	39	20 - 25	1 - 70
Michels & Gibson	65	25 - 75	10 - 1000
Vasilesco	69	0 - 1600	1
Bonilla	88	370 - 1100	1
Sibbitt	111	20 - 500	1 - 70
Lazarre & Vodar	112	25 - 75	1 - 3000
Ross & Brown	113	-50 - 25	35 - 654

these outlets a Bourdon type pressure gauge with a full scale reading of 200 Kg/cm^2 is connected with an Ermeto type valve, providing fine adjustments, between the gauge and the system. The other two outlets are connected, through fine adjustment valves, to the vacuum system required for filling and to the gas bottle used to supply the gas under pressure.

The vacuum system used to reduce the pressure to that required for filling is shown in figure 13. The oil vapour diffusion pump is capable of a vacuum of 5×10^{-6} mm. of mercury but the Pirani gauge, used to indicate the vacuum, only records accurately to 10^{-4} mm. of mercury. The system above may be evacuated to better than this figure in approximately two hours.

The lens ring joints used to seal the viscometer against pressure are not suitable for vacuum. It was found, however, that provided the lens ring joints at each end of the fall tube were backed by "O" rings, a vacuum of better than 0.01 mm. of mercury could be obtained after less than one hour's running. Care had to be taken to ensure that the system was not held at this vacuum for longer than two hours as the "O" rings tended to fail. For vacuum testing and degassing the Pirani gauge head was connected directly to the end of the fall tube.

To fill the viscometer with gas, the valves to the pressure gauge, vacuum system and gas bottle were opened, with the valve on the gas bottle tightly closed. The vacuum equipment was then used to evacuate the system to a pressure of less than 0.03 mm. of mercury and the valve to the vacuum system was closed. The gas bottle valve was opened and gas was allowed to enter the viscometer until atmospheric pressure was attained. The system was again evacuated to 0.03 mm. of mercury and gas allowed to fill the viscometer to the test pressure. The valve to the gas bottle was then closed and the connections to the gas bottle and to the vacuum system were removed.

Viscosity measurements were carried out in the same manner as previously described for steam. The timing circuit used was that shown in figure 11. The nitrogen gas used for these experiments was guaranteed by the suppliers to have impurities

not in excess of those shown in table 6.

TABLE 6

IMPURITIES IN NITROGEN

Gas	Maximum Volumes Million
Oxygen	10
Carbon Dioxide	20
Hydrogen	20

CHAPTER 4

ESTIMATION OF ACCURACY

The factors which limit the accuracy of the determination of the kinematic viscosity of steam at high pressures and temperatures by a transpiration method in general, and by the present apparatus in particular, may conveniently be considered under the following headings:-

1. Deviations from the required conditions of the Hagen-Poiseuille equation.
2. Accuracy of direct measurements.
3. Experimental corrections.
4. Accuracy of properties of fluids required for the evaluation of viscosity.

4.1 The limits of validity of the Hagen-Poiseuille equation are now discussed to ascertain the errors introduced by this particular use of the equation.

(a) Critical Reynolds Number

In his original investigation, Reynolds[115] demonstrated that for viscous flow in a round tube, turbulence could not exist at a value of $\frac{\rho V d}{\eta}$ less than about 2200. This has subsequently been proved by many workers and although laminar flow may exist for flows in a round tube at Reynolds numbers far in excess of this figure, it is desirable to maintain it as low as possible and certainly below 2200. The largest

value of Reynolds number used in the present experiments is 1400 and since this is considerably lower than the critical value it is assumed that no error due to non-laminar flow can arise.

(b) Variations in Capillary Diameter.

Since the bore of the capillary is nominally 0.020 cm. and the tube was manufactured by drawing, it is likely that the capillary will not be exactly round and of exactly the same diameter along its 30 cm. length. It may be subject to conicity or be slightly elliptical at some points.

The apparatus was, however, calibrated with a fluid of known viscosity and it is assumed that, since the flow is well within the laminar range, any disturbance will have the same effect with steam as it had with the calibrating fluid. No appreciable errors are anticipated from this source.

(c) Slip.

A correction for slip at the walls of the tube is necessary where the molecular free path may not be neglected with respect to the radius of the tube. Millikan[116] found that the capillary acts as if its radius were greater than r by an amount 0.874λ, where λ is the mean free path of the fluid molecules. The uncorrected value of viscosity must, therefore, be multiplied by approximately $\left[1 + 4\left(\frac{0.874\lambda}{r}\right)\right]$ and this correction is applied for the calibration with air

where it amounts to 0.33% of the value of the calibration constant.

No correction is applied for steam calculations since with increasing pressure the slip factor decreases and at the lowest steam pressure used, is entirely negligible. No significant error is anticipated from this source.

(d) Effect of the Capillary End.

The assumptions under which the Hagen-Poiseuille equation is formulated require that the flow should have a fully developed parabolic velocity profile along the entire capillary length. In practice this is not possible since a zero boundary layer condition will exist at the capillary inlet and the flow will develop from this condition to the parabolic profile condition. The length of tube along which this development takes place is generally called the transition length. Since the pressure drop in this transition length does not obey the Hagen-Poiseuille law, a correction must be made to make allowance for it.

The existing conception of this correction is that it is made up of two parts. The first is a kinetic energy correction since kinetic energy is required to accelerate the fluid from the zero boundary layer condition to the fully developed one, and cannot be regained at the exit from the tube. This correction is proportional to the velocity squared. The second

correction is caused by the convergence of the flow from the connecting tubes into the capillary. This convergence necessitates differences in velocity between adjacent flow lines and energy is required to overcome the viscous forces involved. This correction is generally known at the Couette correction and involves an increase in the effective length of the capillary.

Several theoretical and experimental determinations of the transition length of a sharp-edged round tube have been made in past years. These have been made both to define the process of transition and to evaluate the corresponding pressure drop. The pressure drop is generally given in the form

$$m \rho u^2$$

and the transition length in the form

$$\left[\frac{x}{d}\right]_{99\%} = \beta Re$$

where x is the distance from the entrance and d the tube radius.

Employing approximate forms of the Navier-Stokes equation with various assumptions Boussinesq[117], Schiller[118], Atkinson and Goldstein[119] and Langhaar[120] have obtained values for the constants m and β in the above expressions. The equations of these authors are graphed in figure 14 in the form of the velocity ratio against $\frac{x}{d\ Re}$. The most complete form of the Navier-Stokes equation is that used by

Langhaar and his results must be considered the most accurate to date. He found that the velocity profile approached the true parabolic form of laminar flow asymptotically and predicted that the centre line velocity would reach 99% of the asymptotic value according to the expression

$$\left[\frac{x}{d}\right]_{99\%\ \text{Inlet}} = 0.0575\ \text{Re}$$

The experimental determinations of Nikuradse[121]; Shapiro and Smith[122] and Reshotko[123] tend to support this result. The reported values for m are given in table 7.

These results show a scatter of some 25% but this can be narrowed by critical appraisal. Schiller's theoretical value was later stated by the same author to be too low and his experimental work is not of sufficient accuracy to be conclusive. Schiller also concluded that bell mouthed ends are essential for viscometric purposes where the kinetic energy correction is of importance. This conclusion was reached after observing the increase in the value of m, and an apparent variation with velocity, with the flattening of rounded ends and the apparent maximum of 1.19 when the flat end state was reached.

The theoretical determination of Hagenbach was based on slightly incorrect assumptions. Wilberforce re-evaluated,

TABLE 7

REPORTED VALUES OF m

No.	Author	Ref.	m		Remarks
1	Hagenbach	124	$\frac{1}{3\sqrt{2}}$	Theory	Error in reasoning
2	Wilberforce	125	1	Theory	Ideal case
3	Couette	26	1	Theory	Ideal case
4	Boussinesq	117	1.12	Theory	
5	Schiller	118	1.08	Theory	Concluded to be low.
6	Schiller	126	1.06 - 1.23	Experiment	
7	Bond	66	1.08		Appears to be value of (s)
8	Riemann	127	1.124	Experiment	
9	Atkinson	119	1.21	Theory	
10	Langhaar	120	1.14	Theory	

using the assumptions of the ideal case, and arrived at m = 1. Couette obtained this value independently in 1890, and also suggested the correction involving an increase in the effective tube length.

Bond[128] investigated the pressure drop thoroughly by experiment and on the basis of his results postulated three regions of flow. For values of $Re < 10$ the kinetic energy

correction was assumed to be zero and the Couette correction became 1.146r. For $10 < Re < 170$ the Couette correction was zero and the value of m varied and had to be obtained from a graph, based on experimental results.
For $170 < Re < 700$ the value of m appeared to be of the order of unity. It is apparent from the experimental points that the scatter is too large to permit an accurate evaluation of m. This may be due to the low values of the length to diameter ratio used in some of the tests. Bond does not appear to be certain of his conclusions since in a later paper[66] he accepts a value of m which appears to be that of Schiller.

Riemann in a range of Reynolds number from 200 to 1010 found m to have the constant value 1.124 ± 0.006 and stated that he did not support the result found by Bond, that m decreases as Reynolds number increases above 700.

With the exception of Couette and Bond, none of these determinations reveal a Couette correction. This may be due to the accuracy of the experiments but it seems more likely that it is due to the conditions under which the experiments were conducted. The general assumption for the evaluation of the transition length is that

$$\frac{\ell}{d\ Re} = \frac{1}{d\ Re} \sum_{x=0}^{x=\ell} \Delta x$$

where Δx is the interval size along the tube length.

According to the kinetic theory this is only valid provided, λ, the mean free path is smaller than Δx. Now if we consider a system where the interval size is smaller than the mean free path, over a part of the transition length, ℓ', then we have

$$\frac{\ell}{d\,Re} = \sum_{x=0}^{x=\ell'} \frac{\Delta x'}{d\,Re} + \sum_{x=\ell'}^{x=\ell} \frac{\Delta x}{d\,Re}$$

$$= \frac{n\,\lambda}{d\,Re} + \sum_{x=\ell'}^{x=\ell} \frac{\Delta x}{d\,Re}$$

$$= \frac{n\,\lambda}{d\,Re} - \sum_{x=0}^{x=\ell''} \frac{\Delta x}{d\,Re} + \sum_{x=0}^{x=\ell} \frac{\Delta x}{d\,Re}$$

and this may be written in the form

$$\frac{\ell}{d\,Re} = \beta + \left[\frac{n\,\lambda}{d\,Re} - \sum_{x=0}^{x=\ell''} \frac{\Delta x}{d\,Re}\right]$$

where β corresponds to the constant 0.0575 determined by Langhaar and n is the number of intervals of distance equal to the mean free path which correspond to the distance $\sum_{0}^{\ell'} \Delta x'$.

This expression is with sufficient accuracy the same as that generally accepted, except for low values of the product d Re and high values of the mean free path. Bond and Couette indicated that at low values of Reynolds number, the pressure drop in the transition length appeared to become proportional to velocity and not to the velocity squared. The experimental phenomena resulting in this interpretation is more successfully

explained by the expression given above for the transition length which shows that for low values of Reynolds number, the length of tube required to ensure 99% fully developed flow may be longer than had previously been postulated. This causes the pressure drop in the inlet length to be a larger proportion of the total pressure drop across a known length of tube - the magnitude of the pressure drop in the transition length remaining the same and proportional to the velocity squared.

The capillaries used for the present experiments had an approximate form of rounded entrances and exits. These were obtained by drilling a 0.025" hole, 1/16" deep and a 0.015" hole, 1/64" deep and attempting to smooth the result into a bell shaped section. Since, in these experiments, the Reynolds number is not sufficiently low or the mean free path sufficiently high, a value of 1.12 has been accepted for m and should be accurate to $\pm$ 3%. The maximum percentage of the total pressure drop which is proportional to the velocity squared is 3% and this introduces a maximum uncertainty in the final viscosity value of $\pm$ 0.09%.

4.2 (a) Measurement of temperature.

The error in the value of temperature accepted as that at which a value of viscosity is measured is due to the following.

(i) The standard thermocouple used to calibrate thermocouple C was guaranteed as accurate to $\pm$ 0.1 C^{o} up to 630 C^{o} and to $\pm$ 0.3 C^{o} above this temperature.

(ii) The calibration of thermocouple C is accepted as accurate to $\pm$ 0.1 C^{o}.

(iii) An interpolation error of $\pm$ 0.1 C^{o}.

(iv) The existing thermocouples were calibrated <u>in situ</u> and this calibration should also be accurate to within $\pm$ 0.1 C^{o}.

(v) The calibration of the existing thermocouples was carried out before the observations at each particular temperature and repeated after the observations had been made. In no case were the results of the calibration before, different from those recorded after the observations by more than 0.5 C^{o}. This is an exception, however, and it seems necessary to consider the accuracy at each isotherm, separately, accepting the mean of the measured values for each thermocouple. The accuracy of measurement is then considered as the difference between the mean and the limits of that thermocouple which indicates the poorest accuracy.

(vi) For each determination of viscosity the thermocouples

1, 2, 3 and 4 were maintained as nearly as possible at the same temperature. The temperature is recorded as the mean of the readings on thermocouples 2 and 3 provided the readings from thermocouples 1 and 4 were not more than 3°C different from that mean. The accuracy is again the difference of the upper and lower values recorded by thermocouples 2 and 3, from their mean.

The measurement of e.m.f. and cold junction temperature are assumed to introduce no significant error and likewise with the temperature of the calibration fluid.

The error in the value of viscosity, caused by an error in temperature varies with the temperature and pressure. The accuracy of temperature measurement has, therefore, been quoted for the temperature alone and has not been included in the evaluated standard deviation of the viscosity values. The accuracy has been given as the extreme case of the arithmetic sum of the errors in the temperature, (i) to (vi), and it is likely that the majority of temperatures are well within the quoted accuracy.

(b) Measurement of Pressure.

The steam pressure was measured, as previously described, by means of a Budenberg type dead weight gauge. The uncertainty of this gauge is a maximum at the highest pressures and

decreases as the pressure decreases. It is better than ± 0.1% at 1000 Kg/cm^2 and ± 0.05% at 200 Kg/cm^2.

The pressure dependence of viscosity is a maximum at the lowest pressure - 0.5% per atmosphere at 200 Kg/cm^2 - and falls to a minimum at the highest pressure. The error in hydrostatic pressure is, therefore, negligible.

No errors are considered for fluctuations in pressure since results were not accepted if a make up of oil was required during or immediately before a timed length.

(c) Mass of mercury pellet.

The mass of mercury used to cause the pressure drop across the capillary was weighed on a chemical beam balance reading to 0.1 mg. The mass of mercury was weighed before and after each test run and for all accepted readings, these values agreed to within 1.0 mg.

Since pellets of approximately 2 gm. were used the mass of mercury is known to better than 0.05% and the corresponding uncertainty in the measured value of viscosity is 0.05% which is negligible.

(d) Measurement of drop tube dimensions.

The volume swept out by the pellet for each timing must be known for the determination of end correction, surface tension drag, compressibility and Reynolds number.

Measurement of length is effected by means of a Pye

cathetometer capable of reading to 0.05 mm. over a length of 500 mm. and 0.005 mm. over a length of 30 mm. The drop lengths are, therefore, known to better than 0.1%.

The area of the drop tube is measured at three points along each drop length by measuring the length of a known mass of mercury - the length of the menisci are also measured. The accuracy of length measurement is again better than 0.1%. The area in each drop length was found to vary by not more than 3% and the swept volumes should, therefore, be accurate to better than this figure. The error from this source will, therefore, be negligible in the corrections for surface tension drag and compressibility. The error in Reynolds number is also of no account since this figure need not be known accurately. The total uncertainty in the end correction is then ± 6% which causes an inaccuracy in viscosity of ± 0.18%.

(e) Times

The apparatus used for measuring the time of fall of the mercury pellet during calibration is capable of an accuracy of ± 1 in 10^5. This accuracy cannot be reproduced in measured times, however, due to irregularities in the bore of the drop tube and to the presence of small quantities of dirt. The standard deviation of all timings recorded, during one pellet run, in the six calibrations of the old drop tube and new capillary used for measurements at 480°C was 0.10% and the

standard deviation of the corresponding timings recorded for two pellet runs was 0.24%. The corresponding standard deviations for the capillary and drop tube used for measurements at 430°C, 380°C and 540°C were 0.07% and 0.15% and for that used at 650°C - and also for measurements with nitrogen - were 0.07% and 0.14%. These figures are obtained by considering the deviations of the recorded timings from the arithmetic mean of these timings, for each separately calculated value of the calibration constant.

The standard deviations for timings recorded for steam viscosity measurements were evaluated in a similar manner to those above but were considered over all accepted results. This yielded values of 0.14% and 0.20% respectively, for the drop tube in the vertical position and in the angled position. Viscosity results were not accepted if the recorded timings showed a deviation of more than 0.5% from the arithmetic mean.

4.3 (a) Surface Tension Drag

The upper meniscus of the falling mercury pellet is less curved than the lower and the effects of capillarity are not equal and opposite. The value of the correction necessary to make allowance for this retardation depends to some extent on the absolute pressure, the fluid in contact with the mercury and the regularity of the drop tube bore. The corrections necessary for the measurements of the viscosity of water at

temperatures in the range 20 - 56°C indicated that the correction does not depend upon the temperature.

The correction represents approximately 10% of the value of the calibration constant, K, and is of the order of 6% of the value of viscosity. The accuracy of its determination depends upon the accuracy of timing and may be evaluated in the following manner.

CALIBRATION - The surface tension drag correction was determined by dividing the mercury pellet into two parts which gives a resultant force due to surface tension of twice that obtained during one pellet runs.

The correction may, therefore, be written in the form

$$\left[1 - \frac{t' - t}{2t' - t}\right]$$

and the error in the second part of this term may be written

$$\log \epsilon = \log (t' - t) - \log (2t' - t)$$

and by differentiation

$$\% \epsilon = \left[\frac{\Delta' t' + \Delta t}{t' - t} - \frac{2\Delta' t' + \Delta t}{2t' - t}\right] \frac{1}{100}$$

$$\doteq \frac{\Delta' t'}{t' - t} \cdot \frac{1}{100}$$

This error has been evaluated for all calibrations of the drop tubes and capillaries to give values of ± 0.23% and ± 0.14%. The first result refers to the old drop tube and capillary and the second to both the '25 cm. drop length' drop tube and

capillary and the '10 cm. drop length' drop tube and capillary. These figures give rise to a negligibly small uncertainty.

VISCOSITY MEASUREMENT - The correction was evaluated by recording timings for the drop tube at a known angle, it being impossible to use the method of two pellets. It has been shown by several authors that the value of the correction does not vary with the angle used and for the present determinations an angle of 52^{o} from the horizontal was employed for both directions of flow. The correction may be written in the form - see Appendix D -

$$\left[1 - \frac{t' \sin\theta - t}{t' - t} - \frac{H - H'}{K W F (t' - t)}\right]$$

In the final evaluation of viscosity, the error due to any likely error in the third term of the above expression is negligible. The error in the second term may be written as

$$\%\epsilon = \left[\frac{0.788 \; ' t' + \Delta t}{0.788 \; t' - t} - \frac{\Delta' t' + \Delta t}{t' - t}\right] \frac{1}{100}$$

and this has been evaluated for steam results to give errors ranging from ± 1.18% to ± 1.23%. The error is, therefore, considered to be ± 1.20% and represents the largest single error in the present determinations of the viscosity of steam.

Measurements carried out on the nitrogen indicated that the standard deviation of the timings was marginally better than for calibration. The possible error in the viscosity of

nitrogen due to surface tension drag was evaluated to give a maximum value of ± 0.37% and a minimum of ± 0.35%. The error is, therefore, considered as ± 0.37%.

(b) Pressure loss in the connecting tubes.

The viscometer was designed in such a manner that this correction was at its practical minimum. The diameter of the drop tube is, however, limited by consideration of possible leakage of water past the mercury pellet and the diameters of connecting tube and pressure vessel are also limited since it is desirable to maintain the volume of the steam space at a minimum. The correction is, therefore, small but not negligible.

The experimental evaluation of the correction by assembling the apparatus in the normal way but without the capillary proved impossible. This was attempted using a mercury pellet of 0.5 gm. and positioning the drop tube at a 45° angle. The resistance to flow in the connecting tubes was so low, however, that the pellet broke up and could not be made to give a measurable time. Since direct measurement had proved impossible and since the above experiment had indicated the small magnitude of the correction it was decided to apply the results of an experiment, carried out by Kjelland-Fosterud, to the apparatus.

This experiment consisted of passing water from a constant head tank, through the apparatus - arranged without the capillary -

and measuring the flow. A water manometer gave the pressure head at inlet and exit from the apparatus. The experiment was carried out with furnace temperatures of 14 C^o and 70°C. The results indicate that the flow was laminar throughout the apparatus.

The pressure loss in the connecting tubes may be given by the expression

$$\Delta p = \Delta p_1 + \Delta p_2 + \Delta p_3$$

where

Δp_1 = loss in part of apparatus containing water = $A \nu_w$

Δp_2 = loss in part of apparatus at transition from water to steam = $B \nu_T$

Δp_3 = loss in part of apparatus containing steam = $C \nu_S$

A, B and C are dimensional constants.

By equating the results at the two temperatures and assuming $B = \frac{20}{65} C$, which is the ratio of their approximate lengths - the cross sectional areas being equal - it was found that

$$A = 4.3 \times 10^6 \text{ w}; \quad B = 1.26 \times 10^6 \text{ w}; \quad C = 4.1 \times 10^6 \text{ w}$$

where w = mass flow through the apparatus.

∴ The viscosity of the steam

$$\nu_s = \frac{\pi d^4}{128 e} \cdot \frac{t}{\pi/4 \, D^2 \, L \, \rho_w} \left[\frac{Wg \, (1 - a/W)}{\pi/4 \, D^2} \left(1 - \frac{\rho_w}{\rho_m}\right) - \frac{\pi}{4} \frac{D^2 L}{t} \rho_w \right.$$

$$\left. (4.3 \nu_w + 1.26 \nu_T + 4.1 \nu_s) \, 10^6 \right]$$

$$= WKtg\ (1 - {}^{a}/W)(\frac{1}{\rho_w} - \frac{1}{\rho_m}) - (\frac{\pi}{4} D^2)^2 L.K.\rho_w\ 10^6$$

$$(4.3\nu_w + 1.26\nu_T + 4.1\nu_s)$$

$$= KtWf\ (1 - {}^{a}/W) - K\rho_w\ (4.3\nu_w + 1.26\nu_T + 4.1\nu_s)10^6(\frac{\pi}{4} D^2)^2 L.$$

For the drop tube used at 380 C°, 430 C° and 540 C°

$$(\frac{\pi}{4} D^2)^2\ L = 0.049$$

which gives, on neglecting the term involving ν_T.

$$\nu_s = \frac{KtWf\ (1 - {}^{a}/W)}{1 + K\rho_w\ 2.01 \times 10^5} - \frac{K\rho_w\nu_w\ 2.11 \times 10^5}{1 + K\rho_w\ 2.01 \times 10^5}$$

$$= KtWf\ (1 - {}^{a}/W)(1 - 6.08 \times 10^{-4}) - 6.48 \times 10^{-4}\ \nu_w$$

to the first approximation.

Within the limits of accuracy considered, the term involving ν_s may also be neglected. The only significant correction required for pressure loss in the connecting tube is that to allow for the pressure loss on the water side of the apparatus and this is sufficiently small that no significant error should be caused by the use of the above expression.

(c) Thermal expansion.

The dimensions of the capillary at room temperature are different from those at steam temperatures. The capillary constant is, therefore, multiplied by the factor $(1 + \Delta t)$ where t is the difference between the steam temperature and the

calibration temperature and Δ is the coefficient of cubical expansion of the platinum 10% rhodium capillary. Since the correction is never significantly greater than 1% of the value of viscosity no error should arise from this source.

(d) Buoyancy.

A correction is required to take account of the upthrust given to the mercury pellet by the working fluid. This correction is in the form $(1 - \rho_f/\rho_m)$ where ρ_f is the density of the fluid, i.e. water and ρ_m is the density of the mercury. For calibration with air the correction is negligible. For steam measurements, however, the value of the buoyance term is finite and must be considered. The values of density are well known and no error is incurred from this source.

(e) Compressibility

The derivation of the Hagen-Poiseuille equation is valid only if the rate of flow of the fluid through the capillary is independent of the position of the mercury pellet in the drop tube. This is true for an incompressible fluid but is not so far a compressible one. In his 1910 contribution, Rankine derived a correction involving an accurate knowledge of the geometry of the system and of the absolute pressure. Since such a knowledge is not always possible he developed a first approximation and proved that for a pellet of less than 5 cm. length in a gas at atmospheric pressure, his

correction represented an error in 1 in 2700. This evaluation was for the particular case where the volume of gas swept out by the pellet is symmetrically placed with respect to the remaining volume of the viscometer.

For the present calibration apparatus we have

V = volume unoccupied by the mercury,

P = steady pressure of the gas when the apparatus is horizontal,

p = difference in pressure caused by the mercury when the apparatus is vertical,

p_1 = pressure and v_1 = volume at any time, behind the mercury pellet,

p_2 = pressure and v_2 = volume at any time, between the mercury pellet and the capillary.

Then

$$V = v_1 + v_2 \quad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

$$p = p_2 - p_1 \quad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

If we assume that there is no change of temperature then

$$PV = p_1 v_1 + p_2 v_2$$

$$= p_1 v_1 + (p + p_1)\, v_2$$

$$= p_1 V + p\, v_2$$

$$\therefore \quad p_1 = P - p + \frac{p v_1}{V}$$

Assume that an element of gas of volume dV_1 passes through the capillary and into the volume v_1 then

$$dV_1 = dv_1 + v_1 \frac{dp_1}{p_1}$$

where dp_1 and dv_1 are the resulting increases in pressure and volume.

$$\therefore \quad dV_1 = \frac{V}{p} \cdot dp_1 + \frac{V}{p} (p_1 - P + p) \frac{dp_1}{p_1} \quad \ldots\ldots\ldots(3)$$

From Meyer's Transpiration formula we have, neglecting slip,

$$dV_1 = \frac{\pi r^4 (p_2^2 - p_1^2)}{16 \eta \ell p_1} dt \quad \ldots\ldots\ldots\ldots\ldots(4)$$

$$= \frac{K (p_2^2 - p_1^2) dt}{2 p_1}$$

$$= \frac{K p (2p_1 + p) dt}{2 p_1} \quad \ldots\ldots\ldots\ldots(5)$$

Equating equations (3) and (5) and writing $\chi = 2p_1 + p$

and $d\chi = 2 dp_1$

we have

$$\frac{V}{p} d\chi \quad (\chi - P) = K p \chi dt$$

Integration gives

$$\frac{V}{p} \left[\chi - P \log \chi\right]_{\chi_1}^{\chi_2} = K p dt$$

where

$$\chi = 2 (P - p + \frac{pv_1}{V}) + p = 2P - p (1 - \frac{2v_1}{V})$$

Supposing t is the time for the upper volume to increase from v_1 to v_1^1 the equation for viscosity is given by

$$2(v_1^1 - v_1) - \frac{PV}{p} \log \left[\frac{1 - \frac{p}{2P}\left(1 - \frac{2v_1^1}{V}\right)}{1 - \frac{p}{2P}\left(1 - \frac{2v_1}{V}\right)} \right] = \frac{\pi r^4 pt}{8\eta l} \quad \ldots.(6)$$

Expansion of the second term according to the Mercater Theorem and substituting $\alpha = \frac{p}{pV}$ gives

$$\frac{1}{\alpha} - \frac{\alpha V}{2}(1 - y) - \frac{\left[\frac{\alpha V}{2}(1 - y)\right]^2}{2} - \frac{\left[\frac{\alpha V}{2}(1 - y)\right]^3}{3} + \frac{\alpha V}{2}(1 - \beta)$$
$$+ \frac{\left[\frac{\alpha V}{2}(1 - \beta)\right]^2}{2} + \frac{\left[\frac{\alpha V}{2}(1 - \beta)\right]^3}{3}$$

where $y = \frac{p}{2P}\left(1 - \frac{2v_1^1}{V}\right)$ and $\beta = \frac{p}{2P}\left(1 - \frac{2v_1}{V}\right)$ and only the first three terms of the expansion are considered. This is reduced and reintroduced into equation 6 to give

$$2(v_1^1 - v_1) - (v_1^1 - v_1) - \frac{p}{2P}(v_1^1 - v_1) + \frac{1}{V}(v_1^2 - v_1^{12}) + \text{etc.}$$

$$= \frac{\pi r^4 pt}{8\eta l}$$

and since $(v_1^1 - v_1) = v$ = volume displaced by the pellet, we have a reduction - to the second approximation -

$$\text{L.H.S.} = v\left\{1 - \frac{p}{2P}\left[\frac{V - (v_1^1 - v_1)}{V} - \frac{p}{2P} + \frac{p}{PV}(v_1^1 + v_1) - \frac{4}{3}\frac{p}{PV^2}(v_1^2 + v_1^1 v_1 + v_1^2)\right]\right\}$$

Now the correction will be a maximum where

(a) $v_1^1 + v_1 \longrightarrow 2V$

(b) $v_1^1 + v_1 \longrightarrow 0$

$\therefore$ considering $v_1^1 \doteq v_1 = V$ we have

(a) L.H.S. $= v\left[1 + \frac{p}{2P} + \frac{4}{3}\ \frac{p^2}{p^2}\right]$

(b) L.H.S. $= v\left[1 + \frac{p}{2P} + \frac{p^2}{4P^2}\right]$

$\therefore$ for a mercury pellet 1 cm. long at a pressure of 75 cm. of mercury we have

(a) $- v\left[1 + \frac{1}{150} + \frac{4}{3}\left(\frac{1}{75}\right)^2\right]$

(b) $- v\left[1 + \frac{1}{150} + \frac{1}{4}\left(\frac{1}{75}\right)^2\right]$

If timings are considered in each direction of pellet flow then the appreciable error of $\frac{1}{150}$ i.e. 0.7% must be allowed for. In the present apparatus only the sum of timings in each direction is considered and the algebraic sum of errors introduced by neglecting compressibility becomes the sum of

$$v\left[1 + 0.0067 + 0.0002\right] = 1.0069\ v$$

and $$v\left[1 - 0.0067 - 0.00004\right] = 0.9933\ v$$

The error is then 2 in 10^5 which is negligible.

It is easily seen that for pressures above atmospheric, the correction becomes even smaller so that no correction is required in the measurement of the viscosity of steam.

4.4 The following properties are used in the evaluation of the kinematic viscosity of steam:-

Dynamic Viscosity of Air

Density of Water

Density of Mercury

Density of Steam

The dynamic viscosity of air was discussed in the review of the literature and involved an error of ± 0.2%. The density of water and of mercury were obtained from "Chemical Engineer's Handbook" [129] and involve no significant error.

The values used for the density of steam in the region near the saturation line are shown in table 8 and were obtained from reference [130].

Values of the density of steam away from the saturation line were obtained from the 1958 Vukalovitch Steam Tables [131].

TABLE 8

DENSITY OF STEAM NEAR THE SATURATION LINE.

Pressure Kg/cm^2	Temperature ^oC	Density gm/cm^3
250	383.0	0.3009
250	383.6	0.2800
300	387.0	0.4792
300	386.1	0.4862
300	390.3	0.4502
400	378.0	0.5970
400	389.0	0.5594
500	386.0	0.6107
500	389.5	0.6018
600	389.0	0.6322
700	388.0	0.6554
800	388.0	0.6724

4.5 TOTAL UNCERTAINTY

From these errors it may be seen that the standard deviation of the calibration constant is ± 0.29% for the drop tube and capillary used at 480°C and ± 0.22% for the remaining two drop tube and capillary assemblies. The standard deviation of the final values of the viscosity of steam is ± 1.45%.

The calibration constant for the old drop tube and capillary was measured six times and the values were found to be consistent within +0.23% and - 0.29% which substantiates the standard deviation given above. The '25 cm. drop length' drop tube and capillary was calibrated four times - less accurate check calibrations were also made - and the values were consistent within + 0.25% and - 0.35%. The '10 cm. drop length' drop tube and capillary was also calibrated four times and yielded results with extreme values, + 0.30% and - 0.38%, with respect to the arithmetic mean. The calibration results for these last two drop tube assemblies appear to indicate that the calibration standard deviation may be slightly optimistic. This does not materially affect the final standard deviation.

The experimental viscosity results appear to substantiate the final standard deviation since with the exception of near critical points the reproducibility of these points is within the standard deviation.

CHAPTER 5

RESULTS

The results listed below are divided into nine sections -

1. Corrected results of Kjelland-Fosterud.
2. Results for water at atmospheric pressure.
3. Results for steam near the 480°C isotherm.
4. Results for steam near the 380°C isotherm.
5. Results for steam near the 430°C isotherm.
6. Results for steam near the 540°C isotherm.
7. Results for steam near the 650°C isotherm.
8. Results for nitrogen at room temperature.
9. Results for nitrogen near the 330°C isotherm.

1. The results obtained by Kjelland-Fosterud at temperatures of about 430°C were recalculated to take into account the probable errors in pressure and temperature. An arithmetical error was found and corrected in the value at 400 Kg/cm^2. These results are shown in Table 1.

Table 1

Temperature °C	Pressure Kg/cm^2	Kinematic Viscosity cm^2/sec. x10^3	Dynamic Viscosity gm/cm.sec. x10^3	Reynolds Number
428	202.5	3.32	0.277	720
426	403	1.26	0.470	810
430	603	1.21	0.644	830
432	603	1.24	0.653	830
430	1004	1.24	0.800	650

The deviation of the value of kinematic viscosity from the true value at the true temperature and pressure should not be more than ± 2.5%. The pressure quoted should not be subject to any inaccuracy but the quoted temperature may be in error by ± 3C° since the thermocouples were not calibrated at the time these observations were made.

2. As a check on the validity of the first calibration of the old short length drop tube and platinum capillary the viscosity of water was measured in the calibration apparatus. The results are shown in table 2.

Table 2.

Temperature °C	Kinematic Viscosity $cm^2/sec. \times 10^3$	Dynamic Viscosity $gm/cm.sec. \times 10^3$	Reynolds Number
56.5	4.89	4.82	28
45.75	6.06	6.03	23
45.25	6.12	6.06	22
34.4	7.33	7.29	19
32.0	7.51	7.47	18
28.5	8.19	8.15	17
20.4	10.01	9.99	13

In the evaluation of these results it was assumed that there was no pressure drop in the connecting tubes. No significant error in the measured temperature should be present and the quoted values of kinematic viscosity should be within ± 1.5% of the true value.

3. The measurements of kinematic viscosity near the 470°C isotherm were made using the short length drop tube - three drop lengths being used in each direction - and the platinum 10% rhodium capillary. The results are tabulated in table 3.

The drop tube and capillary used in this test were not accurately recalibrated since two of the contact wires on the drop tube were unfortunately broken before this could be effected.

TABLE 3.

Temperature °C	Pressure Kg/cm^2	Kinematic Viscosity cm^2/sec. x10^3	Dynamic Viscosity gm/cm.sec. x10^3	Reynolds Number
473.3 ±0.8	300	2.461	0.308	720
477.0 ± 3.0	300	2.504	0.308	730
478.6 ±1.0	400	1.762	0.347	920
473.3 ±3.5	500	1.467	0.456	710
478.0 ±2.0	600	1.289	0.500	880
487.0 ±3.0	700	1.218	0.523	880

A rough calibration was carried out on the remaining drop length and this indicated that there was no change in the capillary constant.

4. The new, 25 cm. drop length, drop tube was carefully calibrated with the platinum-10% rhodium capillary and this was used to obtain the results shown in table 4.

The capillary and drop tube were carefully recalibrated after the above observations had been made. The calibration constant was found not to have altered appreciably.

Table 4.

Temperature °C	Pressure Kg/cm.^{2}	Kinematic Viscosity cm.^{2}/sec. x10^3	Dynamic Viscosity gm/cm.sec. x10^3	Reynolds Number
367.5 ±1.5	200	1.034	0.60	940
370.0 ±2.0	200	1.987 *	0.28	1270
382.9 ±0.9	250	1.095 *	0.330	1350
382.7 ±0.7	250q	1.192	0.359	1400
383.6 ±0.8	250	1.242 *	0.348	1380
387.0 ±2.0	300	1.181	0.566	810
386.1 ±1.5	300	1.181	0.566	1000
390.3 ±0.8	300	1.152	0.518	970
387.1 ±1.0	300	1.164	0.566	1020
378.0 ±1.0	400	1.180	0.704	780
389.0 ±1.0	400	1.156	0.649	790
389.5 ±1.1	400	1.153 *	0.645	860
386.0 ±0.8	500	1.184	0.723	770
389.5 ±1.2	500	1.163	0.700	750
389.0 ±1.1	600	1.177	0.744	690
388.0 ±1.5	700	1.173	0.773	650
388.2 ±1.4	800	1.191	0.800	600

* All results marked thus were evaluated from a set of less than four timing runs in each direction, including one angled run.

5. The same drop tube and capillary and the same calibration constant were used to obtain the results at 430°C which are given in table 5.

Table 5.

Temperature °C	Pressure Kg/cm^2	Kinematic Viscosity cm^2/sec. x10^3	Dynamic Viscosity gm/cm.sec. x10^3	Reynolds Number
433.0 ±1.5	250	2.480 *	0.288	810
423.0 ±1.5	300	1.745	0.323	940
423.0 ±1.5	400	1.235	0.484	880
427.3 ±0.8	500	1.197 *	0.576	820
428.0 ±2.0	500	1.217 *	0.585	820
427.3 ±2.0	500	1.190	0.572	870
426.5 ±2.0	600	1.189	0.656	780
427.7 ±1.0	600	1.195	0.660	770
429.0 ±0.8	700	1.200	0.685	740

The capillary and drop tube were recalibrated after these observations and to within the accuracy of the calibration - ±1% - no deviation from the original calibration constant could be detected.

6. The same drop tube and capillary were used to obtain the following results at 540°C.

Table 6.

Temperature °C	Pressure Kg/cm²	Kinematic Viscosity cm²/sec. x10³	Dynamic Viscosity gm/cm.sec. x10³	Reynolds Number
538.0 ±1.2	400	2.506	0.356	690
538.0 ±1.8	500	1.968 *	0.397	740
536.6 ±0.8	500	1.955 *	0.395	810
537.0 ±0.8	500	1.933	0.391	810
534.6 ±1.1	600	1.688	0.451	690
534.1 ±1.6	700	1.488	0.488	700
534.4 ±1.6	800	1.440 *	0.549	670
538.1 ±0.6	800	1.419	0.529	825
541.0 ±1.0	500	1.958	0.388	910
540.7 ±1.2	700	1.495	0.475	960

Observations for timings etc. were made for the first eight results shown in Table 6 and the drop tube and capillary were then recalibrated. The calibration constant was found to have altered by approximately 5%. A careful recalibration was carried out and a new calibration constant obtained. This new constant was used to evaluate the last two results in table 6.

7. A new '10 cm. drop length' drop tube and a new capillary were used to obtain the results shown in table 7.

Table 7.

Temperature °C	Pressure Kg/cm²	Kinematic Viscosity $cm^2/sec. \times 10^3$	Dynamic Viscosity $gm/cm.sec. \times 10^3$	Reynolds Number
650.0 ±1.6	300	3.758	0.372	560
653.8 ±2.1	400	3.495	0.378	730
654.1 ±0.8	400	3.466	0.377	685
646.3 ±1.7	500	2.735	0.392	890
649.6 ±1.8	500	2.752	0.391	910
650.0 ±0.8	700	2.092	0.444	990
650.0 ±0.9	800	1.900	0.471	1070

Recalibration of the capillary and drop tube indicated that the calibration constant for the above results had not altered.

8. The same drop tube and capillary were used to determine the viscosity of nitrogen at room temperature. These results are shown in table 8.

Table 8

Temperature °C	Pressure Kg/cm^2	Dynamic Viscosity $gm/cm.sec. \times 10^7$	Reynolds Number
16.0	10	1735	187
16.0	20	1746	380
22.5	20	1820	350
17.0	21	1760	370
17.5	21	1768	370
19.0	41	1821	690
15.0	59	1824	1000
17.0	67	1853	1100
19.0	90	1960	1140
19.0	94	1970	1160
19.0	97	1980	1140
17.2	100	1973	1530
17.2	100	1983	1530
23.0	117	2086	1550

9. The same drop tube and capillary were also used to obtain the viscosity of nitrogen at 330°C. These results are shown in table 9.

Table 9

Temperature °C	Pressure Kg/cm.^{2}	Dynamic Viscosity gm/cm.sec. x10^7	Reynolds Number
327.4	13	2955	31
328.0	39	2946	140
328.0	55	2973	180
326.0	58	2941	190
328.2	81	2955	380
328.6	81	2977	380
326.5	100	2956	330

Appendix C shows the readings recorded for one of the original calibrations of the 25 cm. drop length, drop tube and capillary and shown the evaluations of the calibration constant K.

The evaluation of the kinematic viscosity at 300 Kg/cm.^{2} and 423°C is shown in detail in appendix D.

CHAPTER 6

DISCUSSION OF RESULTS.

The discussion of steam results is conveniently divided into two sections, one pertinent to low pressures and the other to high pressures, the temperature range being high in each case.

6.1 Low pressure - high temperature.

The review of literature contained in chapter 2 indicates the acceptible results in this part of the field and these are plotted to a temperature base in figure 15. Experimental results are frequently correlated using an equation of the form predicted by Sutherland. This equation is based on the kinetic theory but the values assigned to the constants are derived from experimental results. The equation may be given in the form

$$\eta = \frac{\alpha \sqrt{T}}{1 + C/T} \qquad (1)$$

where α and C are constants.

Both Shifrin and Bonilla have expressed their results in this form and have recommended values for the constants.

It has been stated by Kestin[40] that Keyes had noticed a marked departure from the Sutherland type of relationship at low temperatures and that Keyes had evolved the equation

$$\eta = \frac{a_1 \sqrt{T}}{1 + C/T \times 10^{-a_2/T}} \qquad (2)$$

where a_1, a_2 and C are constants.

This modification does not appear to be justified since the results shown in figure 15 appear to support the use of the true Sutherland equation - at least up to 900°C.

In the temperature range up to 900°C the viscosity may also be expressed in the form

$$\eta = a + bT \qquad (3)$$

where a and b are constants.

This equation is empirical and the values of a and b are derived from figure 15 to be

$$a = 0.805 \times 10^{-4}$$

$$b = 0.00407 \times 10^{-4}$$

These values correspond to those given by Shifrin and differ from those given by Bonilla. The graph appears to support the author's view that the results of Shifrin are to be preferred to those of Bonilla.

Also shown on figure 15 are the results of Moszynski[39] and Kreiger[132]. The Moszynski values were obtained at pressures from 2 - 8 Kg/cm^2 and should be slightly higher than the corresponding values for atmospheric pressure. Kreiger's values were obtained from a modification of the Stockmayer[133] equation for the energy of interaction between a pair of polar molecules. This equation and the low pressure, low temperature results of Smith, Schugaiev and Timrot were used to predict the viscosity values at higher temperatures.

6.2 High pressure - high temperature

Due to the lack of experimental results a satisfactory equation has not been derived to cover this range. Three equations - and corresponding tabulated viscosity values - have been evolved from existing results. The first of these was that of Timrot who accepted Schirokov's[134] equation for densities not greater than 0.15 gm/cm^3 and who interpolated graphically for higher values of density. The equation given by Schirokov is

$$\eta = \beta \left(P + {}^{a}/_{v^2}\right) \frac{v^2}{v - b} \qquad (4)$$

where P is the pressure and v the specific volume, a and b are Van der Waàls' constants, and β is, in general, a function of temperature and density.

For steam at high temperatures and pressures and for densities not greater than 0.15 gm/cm^3, this may be written in the form

$$\eta = \eta_t \frac{v^2}{(v - b)^2} \qquad (5)$$

where η_t is the dynamic viscosity at the same temperature as η but at atmospheric pressure.

The values of viscosity at atmospheric pressure, accepted by Timrot, are similar to those of Braune and Linke up to 400°C and the value of the constant b was given as 0.75×10^{-3}, the units of dynamic viscosity being Kg.sec.m^{-2}.

Vukalovitch also used Timrot's experimental results as a basis for his correlation, using the same units and the

Schirokov equation in the same form with

$$b = 2.1\ (t - 120)\ x\ 10^{-6}$$

However he derived his values of ηt from the empirical equation

$$\eta t = \eta_o \frac{1 + \frac{C}{273}}{1 + \frac{C}{T}} \sqrt{\frac{T}{273}} \qquad (6)$$

where $\eta_o = 8.17 \times 10^{-6}$ Kg.sec.m^{-2}

and $C = 961$

Discrepancies of up to 5% exist between these two correlations of the same experimental results. The atmospheric pressure results given by equation 6 agree closely with those given by equation 3 and this, together with the inconsistencies found by plotting Timrot's values of kinematic viscosity to a base of temperature in the range 400 - 450°C, suggests that Vukalovitch's correlation is the better.

The results of Sigwart, Jackson and Timrot were used by Hilsenrath and Touloukian[6][135] in their correlation. The results were computed from the Enskog theory using the equations given by Gardner[136]. The final equation may be given in the form

$$\eta = \eta t\ (1 + 0.175\ \rho\ b + 0.8651\ (\rho\ b)^{\frac{2}{}}) \qquad (7)$$

where ρ is the density in gm/cm^3

$$b = \frac{1.783}{M^{1/4}} \left(\frac{\sqrt{T}}{\eta t}\right)^{3/2} \times 10^{-7}$$

M = molecular weight

T is temperature in °K

This equation deviates by more than 10% from the results of Sigwart and Timrot at several points and for this reason does not appear to be reliable.

Since the Schirokov equation is not valid for the greater part of the field covered by the author and since no suitable alternative equation is available, no attempt has been made to provide an equation for the present viscosity measurements. However, the results may be graphically interpolated as shown in figures 16, 17 and 18.

The isotherms shown on the graph of dynamic viscosity against density - figure 18 - have been extrapolated to zero density. The values of dynamic viscosity found at zero density correspond with those found from the graph of dynamic viscosity against temperature - figure 15. It can be seen from figure 18 that the results of Sigwart and Kjelland-Fosterud tend to support the author's results. Timrot's results may be extrapolated in the same way and give reasonable agreement except at 600°C. The gradient of the lines is, however, different from the present author's and the 600°C isotherm extrapolates to give a dynamic viscosity at zero density which is 3% greater than that found from figure 15.

Figure 19 shows the author's results for the dynamic viscosity of water drawn to a base of temperature. The results of Kestin and Swindells and Bingham[137] are shown for comparison.

6.3 Nitrogen

The standard deviation of all timings recorded for measurements on nitrogen with the drop tube in the vertical position was 0.07%. The corresponding standard deviation for the drop tube in the angled position was 0.15%. These standard deviations yield a value of 0.41% when used to evaluate the standard deviation of the final viscosity results in the same manner as was used for steam results. In the assessment of errors for these steam results the fluctuation of the ambient temperature was considered not to affect the final result. In the case of gases this is not true.

The temperature of the laboratory in which all experiments were carried out was subject to a maximum temperature fluctuation of 10°C. In the course of any one day the apparatus temperature did not fluctuate by more than 5°C. The temperature of the viscometer limb inside the furnace was measured using the existing thermocouples and was considered to be the gas temperature, since the furnace insulation would prevent appreciable temperature difference between the gas and the position of the thermocouples. The temperature of the gas in the cold limb could not be accurately measured and is considered to be subject to a maximum uncertainty of $\pm 2\frac{1}{2}$°C. The corresponding uncertainty in the final value of viscosity is ±1% and leads to a standard deviation of ±1.1%.

The fourteen points measured at atmospheric temperature

are shown in figure 20 and it may be seen that ten of these points lie within the quoted standard deviation. This suggests that the standard deviation is in order and supports the standard deviation evaluated for the measured viscosity of steam.

The values for the viscosity of nitrogen near the 330°C isotherm are also subject to the standard deviation of ±1.1%. The measured temperatures may only be considered accurate to ±2°C at best, since thermocouple 2 broke before these results were obtained. The results do, however, indicate that the apparatus is suitable for the measurement of the viscosity of gases at high temperatures provided the ambient temperature is controlled such that the gas temperature in the cold limb does not fluctuate by more than 0.5°C. A thermocouple should be provided with the hot junction inside the cold limb. The apparatus should then be capable of yielding results with a standard deviation of better than ± 0.5%.

6.4 CONCLUSIONS

1. The kinematic viscosity of steam has been measured at temperatures in the range 380 - 650°C and at pressures in the range 200 - 800 Kg/cm^2. The results are lower than those of Timrot in all cases, the difference being substantially near the saturation line and of the order of 5% at higher temperatures. Sigwart's results agree with the author's within the range common to both.

2. The dynamic viscosity of nitrogen has been measured at atmospheric temperature and at pressures in the range 10 - 117 Kg/cm^2. Comparison of these results with those obtained from the literature indicates that the apparatus is not subject to any measurable errors other than those previously considered.

3. The correlation for steam, obtained by extrapolating an isotherm to zero density and comparing the result with that obtained at atmospheric pressure and at the isotherm temperature, depends largely on the experimental results of Bonilla and Shifrin. The results obtained by these two authors differ by 4% rendering an independent check desirable.

4. The range of conditions covered by the present work has not been successfully attempted before and an independent determination is required. All high pressure, high temperature viscosity measurements have been made using the transpiration method and it is desirable that this check be effected using an entirely different principle of measurement, e.g. the oscillating

disc or rotating cylinder.

5. The dynamic viscosity of nitrogen has been measured at pressures in the range 13 - 100 Kg/cm^2 and at temperatures in the region of 327^{o}C. These measurements indicated that an apparatus of the form used, is suitable for the accurate determination of the viscosity of gases at high temperatures and pressures. The viscosity of a large number of gases has not been measured at temperatures above 75^{o}C and at pressures above 100 Kg/cm^2

CHAPTER 7

ROTATING CYLINDER VISCOMETER

7.1 DESIGN REQUIREMENTS

In accordance with the conclusions drawn in chapter 1.5, some of the mechanical problems associated with the rotating cylinder have been investigated and an apparatus has been designed. The design is based upon the following requirements.

(a) The assembly to be capable of measuring the dynamic viscosity of water and steam at pressures up to 500 Kg/cm^2 and at room temperature. The pressure vessel must be suitable for modification to enable measurements to be made over the full pressure range and at temperatures up to 600^{o}C.

(b) The maximum permissible error in the measured value of viscosity to be not greater than $\pm$ 0.1%.

(c) The pressure vessel seals to be of a mechanical nature and to be as few as possible. The tightening of these seals and the effect of pressure on the vessel must not affect the alignment of the suspension and rotating cylinder.

(d) The maximum Reynolds number to be 300 to ensure that no turbulence, or flows in the radial or axial directions, may exist.

(e) The apparatus to be easily assembled and aligned. The alignment of thesuspended cylinder and guard cylinders to be checked before the rotating cylinder is positioned and the

subsequent location of the rotating cylinder to be fixed with respect to the suspended assembly.

7.2 PRESSURE VESSEL

The high pressure vessel was constructed from Firth-Vickers F.C.B.(T) stainless Steel and was designed on a basis of a stress to rupture in 5000 hours at 600°C. and 500 Kg/cm^2. A line diagram of the pressure vessel and stand is shown in figure 21 and an assembly drawing of the pressure vessel, guard and rotating cylinders is shown in figure 22. The apparatus framework will be grouted to a concrete found situated in a temperature controlled room.

From figure 21 it may be seen that the top flange of the pressure vessel is rigidly fixed on the stand, having been levelled with the aid of the jacking screws and a spirit level. The parts of the suspension located by the top flange are then easily accessible and may be aligned optically prior to assembling the pressure parts. The assembly of the main pressure vessel is simplified by substituting two locating rods for two of the eight studs. These rods permit the vessel to be raised and located with respect to the top flange with a minimum time and effort. They also prevent the two locating lengths of the two inch spiggot from being damaged. When the main body has been secured in position by six studs the locating rods are removed and the original studs replaced.

The apparatus was designed to enable the high temperature

modifications to be carried out with a minimum of expense and effort. It is envisaged that these modifications will include the relocation of the window and rotating seal such that they are not subjected to temperatures in excess of 150°C. The present cylinder dimensions may have to be altered and the present stainless steel ball bearings will have to be replaced by bearings of a more suitable type.

For the rotating cylinder to remain a primary instrument the suspension must be calibrated in vacuum to determine the natural period of vibration. At atmospheric temperature, and temperatures up to approximately 100°C, this is easily effected by replacing the main pressure vessel by the vessel shown in figure 23 and the top cap by a brass cap designed to seal on an "O" ring. The vessel shown in figure 23 is fabricated from brass plate, copper pipe and perspex rod, the brass being silver soldered to the copper and the perspex windows sealed by means of "O" rings. Using these vacuum vessels a vacuum of better than 10^{-4} mm. of mercury has been attained with the aid of the pumping system shown in figure 13. The "O" ring joints are unsuitable for temperatures in excess of 100°C and it is hoped that with suitable seals all measurements may be effected in the main vessel, the rotating seal will be disconnected for this purpose and the vacuum connection attached to the vacant screw thread. The vacuum vessel will be used to its maximum temperature since it permits the natural period to be measured for larger angles of swing.

It is also envisaged that some difficulty may be experienced with the suspension wire at temperatures large enough to cause creep of the wire. With this in mind the cylinder dimensions have been based on a Nimonic 90 suspension wire, the creep properties of which are very good. The pressure vessel dimensions were controlled by the cylinder dimensions and hence by the Nimonic 90 wire. It is unlikely that larger cylinders than those designed for the present arrangement will be required for any high temperature modification and the pressure vessel should, therefore, be large enough to incorporate any new suspension system.

7.3 CYLINDER DIMENSIONS

The dimensions of the cylinders are suitable for the measurement of viscosities in the range up to 600°C. and at all pressures. It may be seen from the tables shown in Appendix E that the present cylinders are more suitable for water at atmospheric pressure and steam at the lower pressures. It is unlikely that the required accuracy can be achieved for values of the angle of deflection less than 1.25°, but for smaller angles results can be obtained which are comparable in accuracy to those obtained by transpiration methods. It seems desirable, however, that new cylinders of more suitable dimensions should be constructed for high temperature work.

The error due to any possible eccentricity in the assembly of the suspended cylinder may be calculated to be 0.1% for each 0.0018 cm. out of alignment. This calculation is based on the work of Inglis[138].

The inside diameter of the outer cylinder, the inside and outside diameters of the suspended cylinder and the outside diameter of the guard cylinders are ground and lap finished. All other dimensions are turned or milled. Measurement of all outside diameters was made using Johannson blocks and a "Magna - Gauge" comparator made by Southern Instrument Co. and capable of measurement to 0.00001". These dimensions were checked on a travelling microscope capable of a similar accuracy. No significant deviations from the original measurements were recorded. Internal dimensions were measured with precision micrometer calipers divided into 0.0001" and allowing 0.00001" to be estimated.

The outside diameter of the suspended cylinder was measured at three points along the length, and four diameter measurements were recorded at these points at 45° intervals around the circumference. These results are shown below - other dimensions are given on figure 24. All measurements were made at 25°C.

SUSPENDED CYLINDER DIMENSIONS

TOP	CENTRE	BOTTOM
1.96515	1.96520	1.96520
1.96515	1.96515	1.96525
1.96517	1.96520	1.96525
1.96516	1.96519	1.96525

Overall Length = 3.14600" ± 0.00001

The guard cylinders are 0.750" long and have diametral dimensions corresponding to those of the inner cylinder ± 0.0001".

The material used for the cylinders - 18/8 Niobium stabilised stainless steel - was submitted to a special heat treatment prior to the removal of the final lathe cut. This treatment was found necessary after tests had indicated that cylinders of similar material and dimensions to the suspended cylinder were subject to dimensional instability of up to 0.008" when exposed to temperatures of 500°C for a short time and slowly cooled in air. Further tests showed that a final heat treatment of two hours at 1050°C and slow air cooling reduced the change in dimensions to 0.0002" at the most. All stainless steel viscometer parts, except the main body and flange, were subjected to this treatment.

7.4 SUSPENSION.

The Nimonic 90 wire chosen to support the suspended cylinder is 0.0052" diameter and approximately 20 cm. long. The low temperature properties of the wire are excellent and it is assumed that the high temperature properties are the most suitable available. The bottom wire holder is partly shown in figure 24 and the top collet arrangement is shown in figure 25. The rod which hangs from the suspended cylinder and supports the mirror is also shown in the former diagram.

The collets are designed to permit the wire to be located in the radial and axial directions such that it is exactly on the centre line of the assembly and that the gap between the suspended cylinder and the guard cylinders is the same at each end. Some skill and experience is required to adjust the suspended cylinder in the axial direction but the location in the radial direction is effected automatically on assembling the collets. The use of the 0.003" centering hole also prevents the wire from being locally over-stressed at the points where it enters the collets.

The mirror used to indicate the angle of deflection is optically polished to one wavelength and has the dimension 7 mm. x 5 mm. x 0.5 mm. It is mounted in the groove provided, with a sliding fit. The suspension was statically balanced by removing material from the bottom flat of the mirror holder until no out of balance could be detected. In order that the mirror may be viewed from outside the pressure vessel the drive tube to the rotating cylinder is provided with three 90° slots, each 30° apart, as indicated on figure 22.

The rod which carried the mirror is subject to a torque from the drive tube and allowance must be made for this in the evaluation of viscosity. The correction is, however, small and may be corrected for with no resulting loss in accuracy.

At the higher temperatures where the elastic properties of the wire are less favourable it may prove expedient to manufacture the

suspended cylinder from a lighter material - e,g. titanium - and so decrease the stress in the wire. A slight decrease in the cylinder dimensions may also prove desirable.

7.5 WINDOW

The usual form of quartz window is unsuitable for high pressure use in the presence of water since the water tends to penetrate the quartz at high pressure and to cause layers of quartz to flake off as the pressure is reduced. A suitable material was found in synthetic sapphire and this was supplied in window form by Linde Co., a division of Union Carbide Corporation. The window is an optically finished disc, the flat surfaces being perpendicular to the C axis and guaranteed flat to better than 0.0001". The window seal is of a Bridgman type and is shown in figure 26. This gives a 20° angle of vision. It should be noted that the face of the metal plug in contact with the window when under pressure must be lapped to the same degree of flatness as the window itself.

Since the window will have to be relocated for high temperature measurements the question of the window working under high vacuum does not yet arise although it must be suitable for the low vacuum required for filling purposes. The present design should, however, prove satisfactory for high vacuum use at temperatures not in excess of 100°C if a second "O" ring is introduced on the vacuum side of the window.

The use of a sapphire window and of a compressed fluid where index of refraction is not unity introduced the problem of refraction to the measurement of the angle of deflection. In the present apparatus the problem may be overcome by calculation since the refractive index of the sapphire window is accurately known,[139] as is that for water at atmospheric pressure and temperature. The change in the refractive index of the water with pressure and temperature may be calculated from the semi-empirical equation.

$$\frac{\mu - 1}{\mu_1 - 1} = \frac{v_1}{v}$$

where μ_1 denotes the index of refraction at specific volume v_1 and μ the index of refraction at specific volume v. Using these values it is possible to evaluate the true angle of deflection. From figure 27 it may be shown that

$$\chi' = a \tan 2\alpha + b \tan\beta + c \tan\gamma$$

$$= a \tan 2\alpha + b \tan\left[\sin^{-1} \frac{\sin 2\alpha}{\mu_{w/s}}\right] + c \tan\left[\sin^{-1} \frac{\sin 2\alpha}{\mu_{w/s} \cdot \mu_{s/a}}\right]$$

From this expression χ' may be graphed against the true angle of deflection, 2α, for different values of the index of refraction of the water μ_w. The indices of refraction of sapphire and air, μ_s and μ_a, remain constant.

For the above expression to be accurately evaluated the distances a, b and c must be accurately known and the telescope and scale must be correctly aligned. Dimensions a and b may be measured directly and c may be determined with the aid of two thin

wire plumb lines and a theodolite. The angle of the scale and the telescope position may also be aligned with the aid of plumb lines and a theodolite.

It is intended to measure the angle for both directions of rotation of the outer cylinder and to check the viscosity values by constructing a top seal to carry a rod to the outside of the pressure vessel. This rod will be fitted with a mirror at its top end and a clutch at its bottom end. It can then be used to rotate the deflected suspension system back to the zero position and the true angle of deflection may then be measured directly.

To determine the natural period of the suspension in vacuum no deflection measurements need be recorded. A check on the amplitudes will, however, be recorded to indicate the degree of damping. These measurements may not be considered as accurate absolute measurements since the perspex window of the vacuum vessel will act as a cylindrical lens.

7.6 SEALS AND DRIVE.

The main pressure vessel seal is effected by means of a fully annealed nickel ring, 1/8" square section. It is hoped that for temperatures higher than "O" rings and the vessel shown in figure 23, can be expected to withstand, the nickel ring will withstand the maximum pressure and permit a vacuum of better than 10^{-3} mm. of mercury to be reached. The ring should provide some degree of self sealing and not, therefore, require large tightening torques to be applied to the studs. This form of seal is used in the atomic

energy industry and is reported to have been successful for vacuum of the order of 10^{-3} mm. of mercury while remaining suitable for operation at 3500 lb/in.^{2} and 350°C.

In the event of this seal requiring excessively large tightening torques or not being suitable for the vacuum required, the apparatus may be modified to permit the use of a wedge ring seal of the form suggested by Niemeier.(140) This form of seal may be designed to require a minimum of initial tightening and experiment has shown that it is capable of supporting a vacuum of better than 10^{-3} mm. of mercury.

With the exception of the window and rotating seal all other joints are effected by means of ball and cone or lens ring seals. Experience with the Rankine viscometer has shown that these seals are capable, with careful machining, of supporting the required vacuum. To reduce the time required to attain the vacuum a brass cap has been constructed to replace the stainless-steel cap of the pressure vessel. This brass cap seals on an "O" ring.

The drive to the rotating cylinder may be effected by means of two magnets, one inside and one outside the pressure vessel. Alternatively a drive shaft may be used and this involves a rotating seal. The magnetic drive is preferable since there can be no possibility of leakage of water from the viscometer and a number of rough experimental tests were carried out to determine if this form of drive is practical. These tests indicated that a C type magnet - of the maximum strength available - is not a practical

proposition if it is to rotate about its own axis. It may, however, be used if it holds a second magnet inside the pressure vessel and at a radius of not less than 4" from the centre line of the pressure vessel. The thickness of the stainless steel and of the air gap between the two magnets must be maintained at a minimum.

The mechanical design of a magnetic drive is not suitable for the high temperature conversion envisaged and it was decided to attempt to construct a rotating seal which would be more suitable for modification. The final design of this seal is shown in figure 28. Tests in this laboratory have indicated that the maximum starting torque required for this assembly is 4 lb.ft. - this figure is likely to be considerably reduced when the teflon plug is correctly seated. Leakage of water past the seal at maximum pressure is likely to be negligible and any make up necessary should be easily provided by the stroke of a dead weight gauge. The seal is also self lubricating and should not be subject to overheating at the low speeds envisaged.

A synchronous motor driven from the 250 V.a.c., 50 c/s mains will be used initially to provide the constant speed drive. The required speed of rotation may be attained by means of a worm reduction and a selection of spur gears. A more refined method of speed control may be introduced at a later date and it is envisaged that this will take the form of a synchronous motor driven from a constant frequency source.

7.7 BEARINGS

For temperatures up to 150°C it is possible to use commercial ball races and these have been incorporated in the present design. The apparatus is, however, easily modificable for bearings more suitable for high temperature steam conditions and tests to determine such a bearing are described in Appendix F.

7.8 PRESSURE AND TEMPERATURE MEASUREMENT

The pressure will be raised, maintained and measured in the same manner as was used for the Rankine viscometer, the rubber diaphragm being replaced by metal bellows of suitable capacity. The connection to the pressure vessel is by means of one of the 0.150" diameter holes which pass through the top flange and enter the main vessel on the outside of the rotating cylinder. Any water make up entering the vessel at this point should have the minimum effect on the flow between the rotating and suspended cylinders.

Provision has been made in the apparatus for four thermocouples. One immediately above the top collets and three, 90° apart, situated in the annular gap between the pressure vessel and rotating cylinder. Closed end stainless steel sheathes have been made to hold 30 s.w.g. thermocouples and are readily accessible for calibration. For low temperature measurements the thermocouples will be iron-constantan and will be accurately calibrated against a platinum resistance thermometer.

7.9 PERIOD MEASUREMENT

Period measurement is necessary to evaluate the moment of

inertia of the suspended cylinder, rod and attachments and to determine the natural period of oscillation of the complete assembly. The moment of inertia of the suspension may be calculated from the known dimensions but the total value must be checked by experiment. This may be carried out in the viscometer with the vacuum vessel connected and both guard cylinders omitted. To measure the moment of inertia it is necessary to construct an object of moment of inertia and mass similar to that of the suspended system. This object must be of simple shape such that its moment of inertia may be accurately calculated from its dimensions. The most suitable shape appears to be a disc with a central hole such that it will push fit into the lower diameter of the bottom collet holder.

The natural period T_1, of the assembled suspension is then measured in vacuum, the vacuum released and the vacuum vessel and rod removed. The suspended cylinder may then be raised some 2.5 cm. and held in an out of the way position by means of tape. The disc of known moment of inertia may then be push fitted onto the collet holder, the vacuum vessel replaced and the natural period T_2 of the new suspension measured. Since we have

$$I_1 = I_2 \frac{T_1^2}{T_2^2}$$

and the moment of inertia of the collet holder may be calculated with sufficient accuracy, the moment of inertia of the suspension may be determined. The length of wire used is of little importance provided it is the same for the determination of both T_1 and T_2.

The natural period of oscillation of the suspension used for viscosity measurements must be measured with the top guard cylinder in position so that the suspension need not be altered between the measurement of period and the measurement of viscosity. The arrangement intended for period measurement is shown on figure 29a and the circuit diagram shown in figure 29b indicates the method of pulsing the counter. This timing arrangement can be used to record timings for one period or for a number of periods. It is likely that the photocell output will require to be amplified and shaped in order that the relay will close positively at the same light intensity for each passing of the light source. The circuit, however, has been proved in these laboratories to work in principle and it remains to refine it.

7.10 EVALUATION OF VISCOSITY.

With all cylinder dimensions, the moment of inertia of the suspension and the natural period known, it remains to measure the angle of deflection of the inner cylinder for a known speed of rotation of the outer cylinder and the dynamic viscosity of the fluid under consideration may be evaluated from the expression given in chapter 1.2 and the equation

$$T_o = 2\pi\sqrt{\frac{I\theta}{T}}$$

where T_o is the natural period and T the restraining torque.

ACKNOWLEDGEMENT

The work described in this thesis forms part of a programme of research into the properties of steam which is supported by the Electricity Supply Research Council and is being carried out at the Mechanical Engineering Research Annexe, The University of Glasgow.

The author is indebted to Professor James Small, James Watt Professor of Mechanical Engineering, in whose department he has been privileged to carry out his research and to his colleagues of the Research Annexe who have assisted him in many ways.

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APPENDIX A

CALIBRATION OF THERMOCOUPLES

(e.m.f. Values in Microvolts)

Results of Test on "Probe" Thermocouple.

Standard Thermocouple.	204	282	426	579°C
Probe Thermocouple	204	280	420	569°C.

Calibration Chart for Thermocouple C.

°C	100	200	300	400	500	600	700
0	640	1431	2311	3245	4215	5221	6261
10	714	1516	2402	3340	4314	5324	6367
20	788	1601	2495	3436	4414	5426	6473
30	864	1687	2587	3533	4513	5530	6580
40	942	1774	2680	3629	4614	5633	6687
50	1021	1862	2774	3725	4714	5737	6794
60	1101	1951	2868	3822	4815	5841	6902
70	1182	2040	2962	3920	4916	5945	7009
80	1264	2130	3055	4018	5017	6050	
90	1347	2220	3150	4116	5119	6156	
100	1431	2311	3245	4215	5221	6261	

Calibration of Existing Thermocouples using Thermocouple C.

480°C

A	1.	2.	3.	4.	5.
	4238	4201	4190	4217	4206
		4203	4190	4217	
Thermocouple C.		3997	4003	4026	
		3999	4005	4028	
	Both cold junction temperatures = 0.0°C				
B.	4235	4200	4192	4229	
		4200	4197	4231	4191
Thermocouple C.		3997	4005	4036	
		3997	4010	4038	
	Both cold junction temperatures = 0.0°C				
δA.		0.9°C	3.2°C	3.4°C	
δB.		0.9°C	3.2°C	3.2°C	

380°C

A.	1.	2.		3.		4.	5.
	3220	3173	C.J.18°C	3166	17.8°C	3148	18°C 3105
		3173		3165		3148	
Thermocouple C.		3149		3150		3142	
		3150		3152		3142	
B.	3141	3081	C.J.28.1°C.	3062	28°C	3051	28°C 3020
		3078		3060		3051	
Thermocouple C.		3121		3112		3109	
		3119		3112		3111	
δA.		2.0°C		3.0°C		3.7°C	
δB.		1.9°C		3.4°C		4.2°C	

430°C.

A.	1.	2.		3.		4.		5.
	3550	3508	23.0°C	3502	23.1°C	3517	23.1°C	3525
		3510		3504		3519		
Thermocouple C		3485		3490		3512		
		3489		3494		3514		
B.	3624	3600	28.2°C	3625	28.5°C	3609	28.6°C	3661
		3603		3615		3603		
Thermocouple C		3603		3635		3626		
		3593		3622		3623		
δA.		2.1°C		3.1°C		4.0°C		
δB.		1.8°C		3.0°C		4.0°C		

540°C

A.	1.	2.		3.		4.		5.
	4796	4747	22.8°C	4731	22.8°C	4740	22.8°C	4705
		4740		4723		4731		
Thermocouple C		4622		4620		4631		
		4616		4613		4623		
B.	4800	4732	14.6°C	4715	14.6°C	4751	15.0°C	4750
		4720		4700		4746		
Thermocouple C		4570		4569		4599		
		4564		4562		4597		
δA.		3.0°C		4.2°C		4.4°C		
δB.		3.1°C		4.2°C		4.0°C		

680°C

	1.		2.		3.		4.		5.	
A.	6361		6282		6260		6286		6302	
	6361	21.6°C	6282	21.6°C	6260	21.6°C	6284	21.7°C	6302	21.7°C
Thermocouple C.										
	6032		6013		6000		6017		6032	
	6032		6013		6000		6017		6032	
B.	6338		6276		6271		6289		6301	
	6338	20.1°C	6276	20.1°C	6271	20.1°C	6289	20.1°C	6301	20.1°C
Thermocouple C.										
	6001		6007		6000		6011		6027	
	6001		6007		6000		6011		6027	
δ_A	-0.4°C		4.7°C		5.0°C.		4.6°C		4.6°C	
δ_B	-0.5°C		4.6°C		5.1°C		4.4°C		4.9°C	

330°C

	1.		2.		3.		4.		5.	
A.	2449		2380		2386		2393		2411	
	2449	26.9°C	2380	26.9°C	2386	26.9°C	2391	26.9°C	2411	26.9°C
Thermocouple C										
			2464		2469		2477			
			2464		2469		2477			
B.	2590		--		2502		2510		2510	
	2590	21.0°C	--		2502	21.0°C	2510	21.0°C	2510	21.0°C
Thermocouple C										
			2567		2550		2556			
			2567		2550		2556			
δ_A			2.1°C		1.9°C		2.2°C			
δ_B			-		2.6°C		2.4°C			

In all cases the temperature of the cold junction of thermocouple C is maintained at 0°C.

Reading A refers to the calibration made prior to the measurement of viscosity at that isotherm and reading B, to that made after the viscosity measurements.

APPENDIX B

CALIBRATION OF EXISTING PRESSURE BALANCE

Nominal Pressure Kg/cm^2	A	B	C
200	13197	13036	12903
300	19650	19489	19354
400	26102	25893	25806
500	32540	32337	32257
600	39000	38795	38708
700	45450	45189	45160
800	51903	51640	51612
900	58357	58089	58063

A - Mass in grams added to original dead weight gauge to give pressures quoted by Kjelland-Fosterud.

B - Mass in grams added to original dead weight gauge to balance the new gauge tester.

C - Mass in grams theoretically necessary to give nominal pressure - based on nominal piston area.

It appears, therefore, that the nominal pressures recorded by Kjelland-Fosterud are in error as shown below..

Nominal Pressure Kg/cm^2	200	300	400	500	600	700	800	900
True Pressure Kg/cm^2	202.5	302.5	403.2	503.1	603.2	704.1	804.1	904.1

APPENDIX C

SPECIMEN CALIBRATION OF CAPILLARY AND DROP TUBE.

3rd Calibration run on 25 cm. Length Drop Tube and New Capillary

Date	Pellet Weight gm.	Temperature	Time from Capillary sec.	Time to Capillary sec.	t
6.8.59	2.1197	19.9	14.6831	15.8814	30.5645
			14.6809	15.8843	30.5652
			14.6741	15.8831	30.5572
			14.6629	15.8666	30.5295
			14.6814	15.8585	30.5399
		20.0	14.6789	15.8543	30.5332
	*Two pellet runs		15.9796	17.1713	33.1509
			15.9973	17.1659	33.1632
			14.6817	15.8623	30.5440
			14.6843	15.8619	30.5462
			14.6792	15.8672	30.5464
			14.6871	15.8659	30.5530
	*Two pellet runs		15.9919	17.1706	33.1625
		20.0	15.9864	17.1775	33.1639

* The pellet is split into two to permit the drag correction to be determined.

Average value of t = 30.5479 seconds.

Average values of t^1 = 33.1601 seconds.

Correction for surface tension drag is

$$(1 - {}^{a}/_{w}) = 1 - \frac{33.1601 - 30.5479}{2 \times 33.1601 - 30.5479} = 0.9270$$

Slip factor = 1.0033

Dynamic viscosity of air at 20.0 C^{o}, $\eta_{20.0}$ = 1816.6 gm/cm.sec.

∴ Calibration constant K

$$= \frac{1816.6}{2.1197 \times 30.5479 \times 0.9270 \times 980.6 \times 1.0033}$$

$$= 30.76 \times 10^{-10}$$

APPENDIX D

SPECIMEN VISCOSITY EVALUATION

The evaluation of kinematic viscosity of steam is made from the equation

$$\nu = KtWF\,(1 - {}^{a}/_{w}) - 0.0068 \times 10^{-3} - H$$

where

K = calibration constant.

t = average of the sum of the timings in each direction.

W = weight of mercury pellet.

$F = (1 + \Delta t)({}^{1}/\rho_{w} - {}^{1}/\rho_{m})\ 980.6$

$(1 - {}^{a}/_{w})$ = correction for surface tension drag

$$= 1 - \frac{t^{1} \times \sin\theta - t}{t^{1} - t} - \frac{H - H^{1}}{KWF(t^{1} - t)}$$

where H^{1} is the end correction corresponding to time t^{1}

and H is the end correction corresponding to time t

$$= 1 - \frac{t^{1} \sin 0 - t}{t^{1} - t} - \frac{1.240 \times 10^{-3}}{KtWF_{s}\, t^{1}_{TO}}$$

H = end correction

$$= \frac{M \rho_{w} Q}{8\pi l \rho_{s}} = 1.12 \frac{\rho_{w}}{\rho_{s}} \frac{D^{2}L}{32\ell t} = \frac{1.24 \times 10^{-3}}{\rho_{s}\ t_{TO}}$$

∴ To obtain a value for the kinematic viscosity at 300 Kg/cm^{2} and 423 C^{o} we have

$K = 30.74 \times 10^{-10}$

$t = 343.88$

$t^{1} = 438.02$

$$F = (1 + 3 \times 8.9 \times 10^{-6} \times 405)\ (0.9884 - 0.0738)\ 980.6$$

$$= 906.5$$

$$\therefore KtWF = 1.846 \times 10^{-3}$$

$$(1 - {}^{a}/_{w}) = 1 - \frac{438.02 \times 0.788 - 343.88}{438.02 - 343.88} - \frac{1.24}{1.846} \times \frac{5.41}{218.9}$$

$$= 0.9698$$

$$H = 0.039 \times 10^{-3}$$

$$\therefore \nu = (1.846 \times 0.9698 - 0.0068 - 0.039)\ 10^{-3}$$

$$= 1.745 \times 10^{-3}\ cm^2/sec.$$

APPENDIX E

ROTATING CYLINDER DESIGN

The design of the rotating cylinder viscometer is based on the equations

$$T = 4\pi L \eta w \frac{a^2 b^2}{a^2 - b^2} \quad \text{.....1.}$$

$$T = \frac{981\,\theta\,G}{\ell} \cdot \frac{\pi d^4}{32} \quad \text{.....2.}$$

$$Re = \frac{w\,(a - b)a}{\nu} \quad \text{.....3}$$

where

T = torque

L = length of suspended cylinder

w = speed of rotation of rotating cylinder.

a = inner radius of rotating cylinder

b = outer radius of suspended cylinder

θ = angular deflection of suspended cylinder

d = diameter of suspension wire

G = modulus of torsion of suspension wire

ℓ = length of suspension wire.

These equations and the choice of a Nimonic 90 suspension wire 0.0052" diameter led to the conclusion that the most suitable cylinder dimensions are

a = 2.75 cm. b = 2.5 cm. L = 8.0 cm.

The wire diameter was initially chosen to maintain the pressure vessel at a reasonable size and the intended length of suspension wire is 20 cm.

The angles of deflection of the suspended cylinder for given speeds of rotation of the outer cylinder are listed below for water and steam at various conditions of temperature and pressure. The angle of deflection of the suspended cylinder is limited to 10° by the mechanical design of the window and the need to measure the angle for both directions of rotation. The maximum allowable value of Reynolds number is considered to be 300.

WATER AT ATMOSPHERIC PRESSURE

Temperature °C	G $Kg/cm^2 \times 10^{-4}$	η gm/cm.sec.	Re	N rev/min.	θ degrees
20	87.9	0.010	35	5	9.89
40	87.2	0.0064	35	3	9.05
60	86.5	0.0047	30	2	8.11
80	85.8	0.0037	40	2	10.2
100	85.1	0.0028	50	1.5	10.1

STEAM AND SUPERCRITICAL WATER

Temperature : $200^{\circ}C$: $G = 83.7 \times 10^4$

Pressure Kg/cm^2	η gm/cm.sec. x 10^3	ν $cm^2/sec. \times 10^3$	Re	N rev/min.	θ degrees
1	0.161	355	20	100	2.88
100	1.40	1.62	268	6	1.49
300	1.46	1.65	261	6	1.56
500	1.50	1.67	259	6	1.57

Temperature : 300°C : G = 80.8 x 10^4

1	0.200	538	13	100	3.68
100	0.920	1.28	281	5	0.84
300	0.971	1.3	277	5	0.90
500	1.01	1.3	277	5	0.93

Temperature: 400°C : G = 79.4 x 10^4

1	0.239	756	10	100	4.48
100	0.249	6.76	210	20	0.93
300	0.451	1.36	265	5	0.42
500	0.675	1.25	289	5	0.63

Temperature: 500°C : G = 78.7 x 10^4

1	0.277	1008	7	100	5.20
100	0.290	9.73	297	40	2.18
300	0.334	2.97	291	12	0.76
500	0.405	1.71	295	7	0.53

Temperature: 600°C : G = 75.6 x 10^4

1	0.315	1292	6	100	6.18
100	0.331	13.0	276	50	3.25
300	0.377	4.38	296	18	1.33
500	0.379	2.50	287	10	0.75

All calculations are based on the cylinder dimensions at room temperature.

APPENDIX F

HIGH TEMPERATURE BEARING TESTS

In order to determine the most suitable type of bearing to run in an atmosphere of steam at temperatures up to 600°C, a series of tests were carried out. The tests were designed to meet the following requirements of the viscometer.

(a) To maintain bearing friction at a minimum without using any form of lubrication other than the steam itself.

(b) To ensure that the rotating cylinder is located in the radial direction to a limit of 0.00025" on its diameter.

(c) To permit the apparatus to be easily assembled.

The apparatus shown in figure 30 was constructed and permitted bearings with a maximum diameter of 60 mm. to be tested at temperatures up to 750°C. The temperature was measured with a moveable chromel-alumel thermocouple and the furnace controlled by means of a variable transformer. The furnace was 14" long, with Sindanyo end pieces to minimise end losses, and a series of tests indicated that the temperature over the centre 6" of the furnace was constant to ± 3 °C at temperatures up to 800°C. In all cases the bearing under test was located in the centre of the furnace.

It was considered sufficient for these tests to use an atmosphere of air and to assume that the most satisfactory bearing in air would also be the most satisfactory in steam. The following tests have been carried out.

1. STAINLESS STEEL ROLLERS

In an apparatus containing steam at advanced conditions it is desirable to have as few different materials as possible. It was, therefore, decided to attempt first the use of stainless steel rollers running free in a carefully machined slot in the inner retaining ring. 18/8 niobium stabilised stainless steel was used for all parts. It was known that this type of material is liable to failure due to 'pick up' but it was hoped that careful machining would obviate this.

Twenty-four rollers 5 mm. long and 0.1969" ± 0.000075 diameter were manufactured and lap finished. The outer cylinder was ground finished to a diameter of 1.7222" ± 0.0001 and the inner ring to $1.3280"\ ^{+0.0000}_{-0.0001}$. The assembly is shown in figure 31a.

The test apparatus was assembled and the outer cylinder rotated at 40 r.p.m. The temperature was slowly increased and the bearing continued to run satisfactorily for approximately 60 hours until a temperature of 380°C was reached. At this temperature the motor current, which was being measured on a d.c. ammeter, began to fluctuate during each revolution indicating that the bearing was sticking.

The apparatus was dismantled and examination of the flat bottom of the rollers showed that small particles of metal had rubbed off. These particles appeared to have worked into the rolling surfaces and became embedded, thereby causing sticking.

2. SYNTHETIC SAPPHIRE DISC.

The properties of synthetic sapphire indicate that it should be an excellent bearing material. It is unaffected by steam at high temperature, can be polished to very fine limits and has a hardness of 9 on Moh's scale. Its coefficient of mean linear thermal expension is of the order of 6×10^{-6} per ^{o}C compared to 16×10^{-6} per ^{o}C for stainless steel.

The large difference in coefficient of thermal expansion of sapphire and stainless steel precludes the use of a sapphire disc for the viscometer bearing. Since a disc was readily obtainable and easily incorporated in the experimental apparatus it was decided to test the sapphire as a bearing surface at high temperature. The arrangement permitted the bearing to be used at $400^{o}C$ with a 0.001" radial clearance between the outer cylinder and the disc. Gold washers were used in the assembly to ensure that tightening of the securing bolt did not crack the disc.

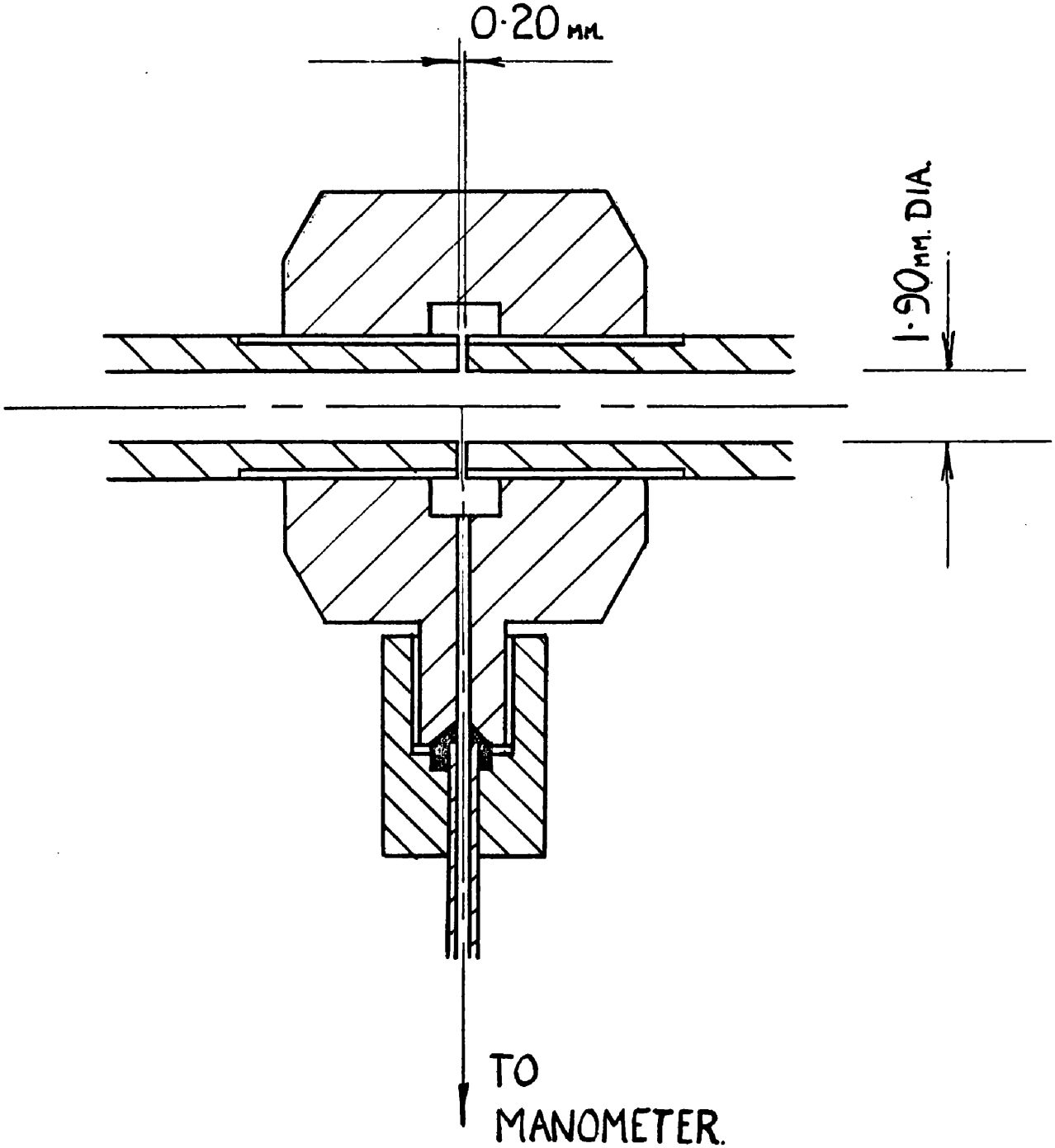
The apparatus was assembled and again set to run at 40 r.p.m., the furnace temperature having been set at $400^{o}C$. The bearing functioned satisfactorily for the ten hours test period.

3. SYNTHETIC SAPPHIRE PADS.

To overcome the problem caused by the thermal expansion of the sapphire, a new bearing was constructed using pads of sapphire as shown in figure 31b. It was found that the assembly of this arrangement was difficult due to the sharp edges of the

pads which tended to tear the metal of the outer cylinder. The apparatus was eventually assembled and ran satisfactorily at 40 r.p.m. for a short time at a temperature of 400°C. The apparatus was dismantled at this point to check the assembly and on re-entering the pads, two were broken.

Recent information on high temperature bearings indicates that sapphire balls, tool steel balls and tool steel rollers can be used in steam at high temperature. These require that the outer and inner cylinders be coated with "Stellite" to a depth of 0.025" to give a Rockwell hardness of 60 - 65. Tests carried out by Linde Co. indicate that the sapphire balls give the lowest coefficient of friction and these balls appear to offer the most satisfactory solution.



SPEYERER'S CONNECTION FIGURE 1

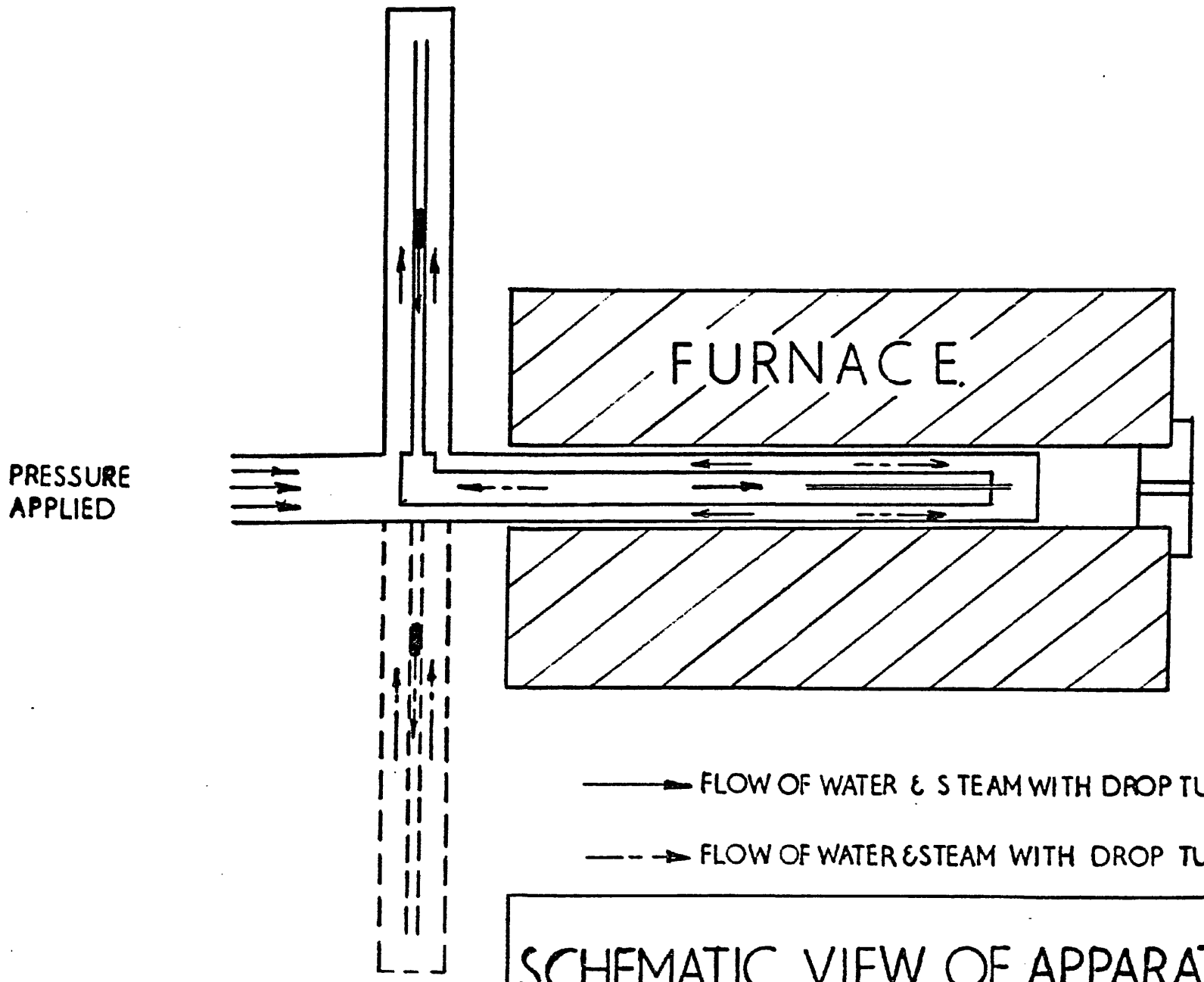
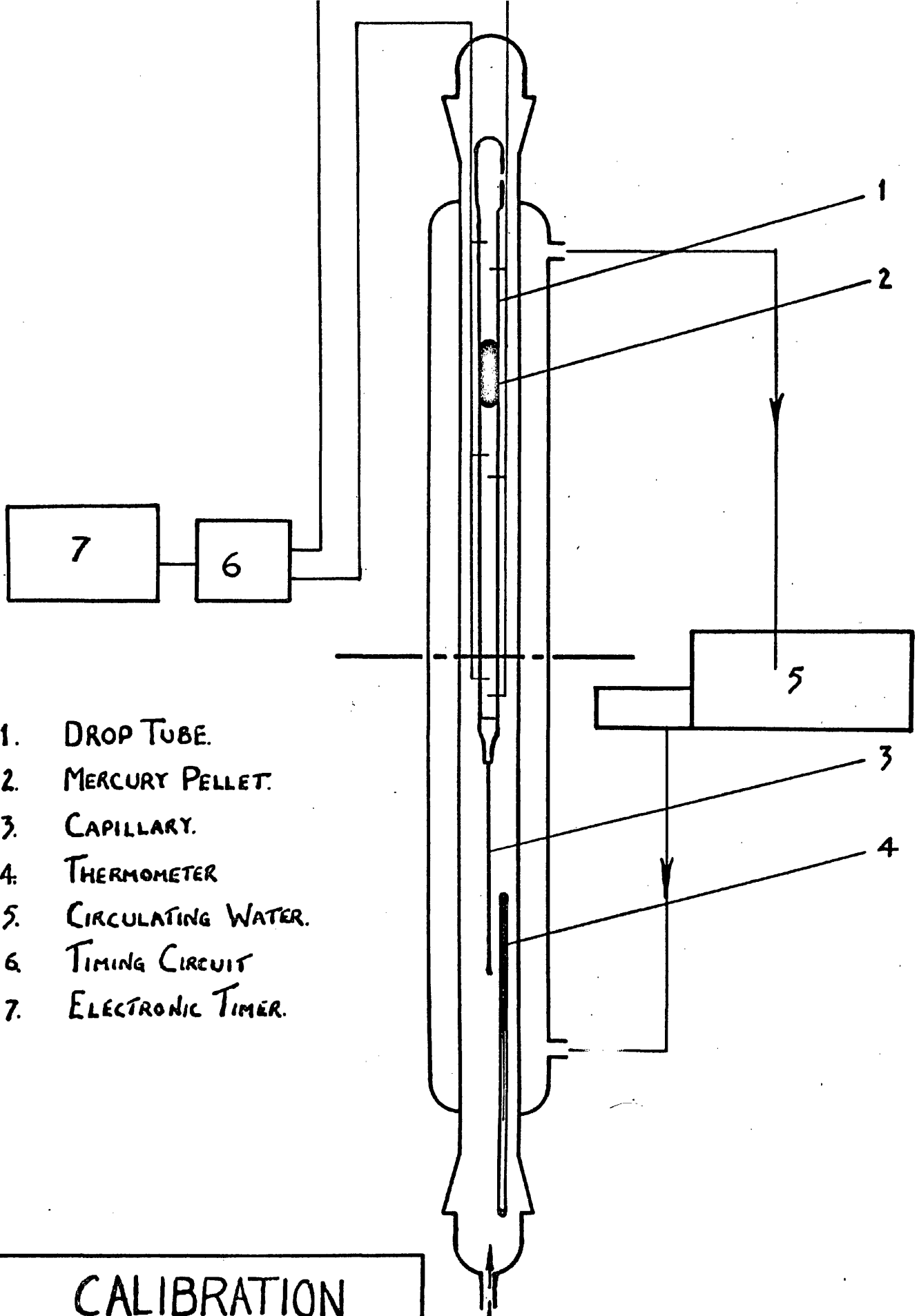


FIGURE 2

SCHEMATIC VIEW OF APPARATUS

NOT TO SCALE.

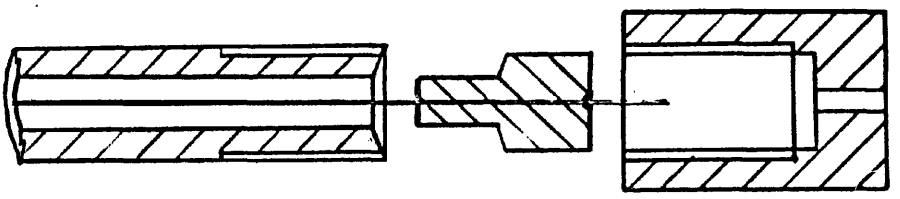
21.3.60



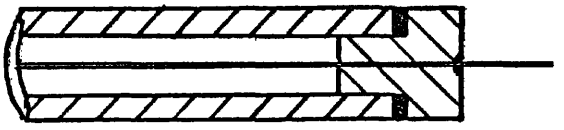
1. DROP TUBE.
2. MERCURY PELLET.
3. CAPILLARY.
4. THERMOMETER
5. CIRCULATING WATER.
6. TIMING CIRCUIT
7. ELECTRONIC TIMER.

CALIBRATION APPARATUS.

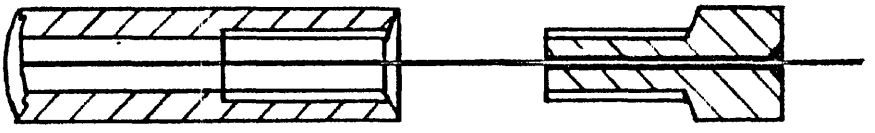
FIGURE 3



a.



b.



c.

CAPILLARY JOINTS

FIGURE 4

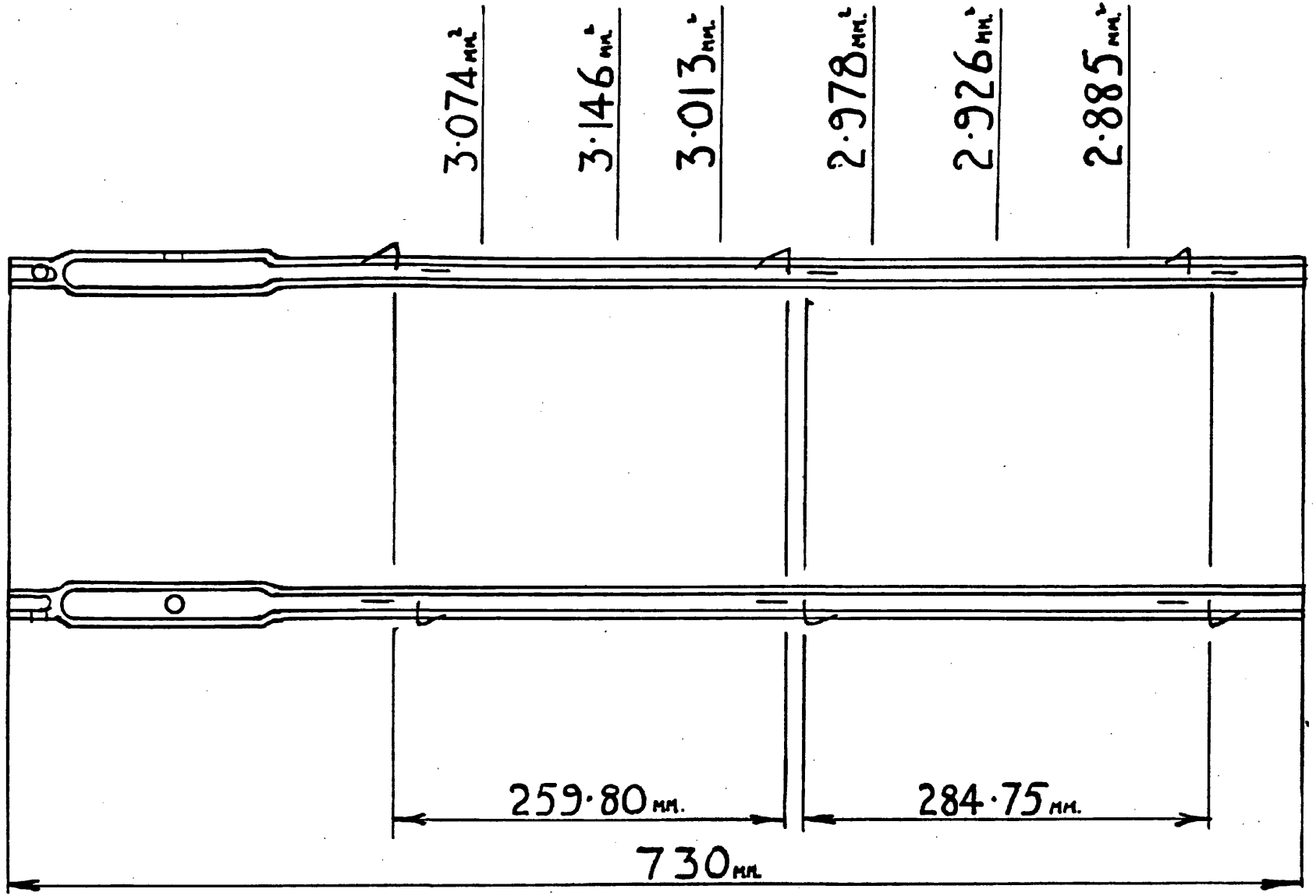


FIGURE 7

DROP TUBE.

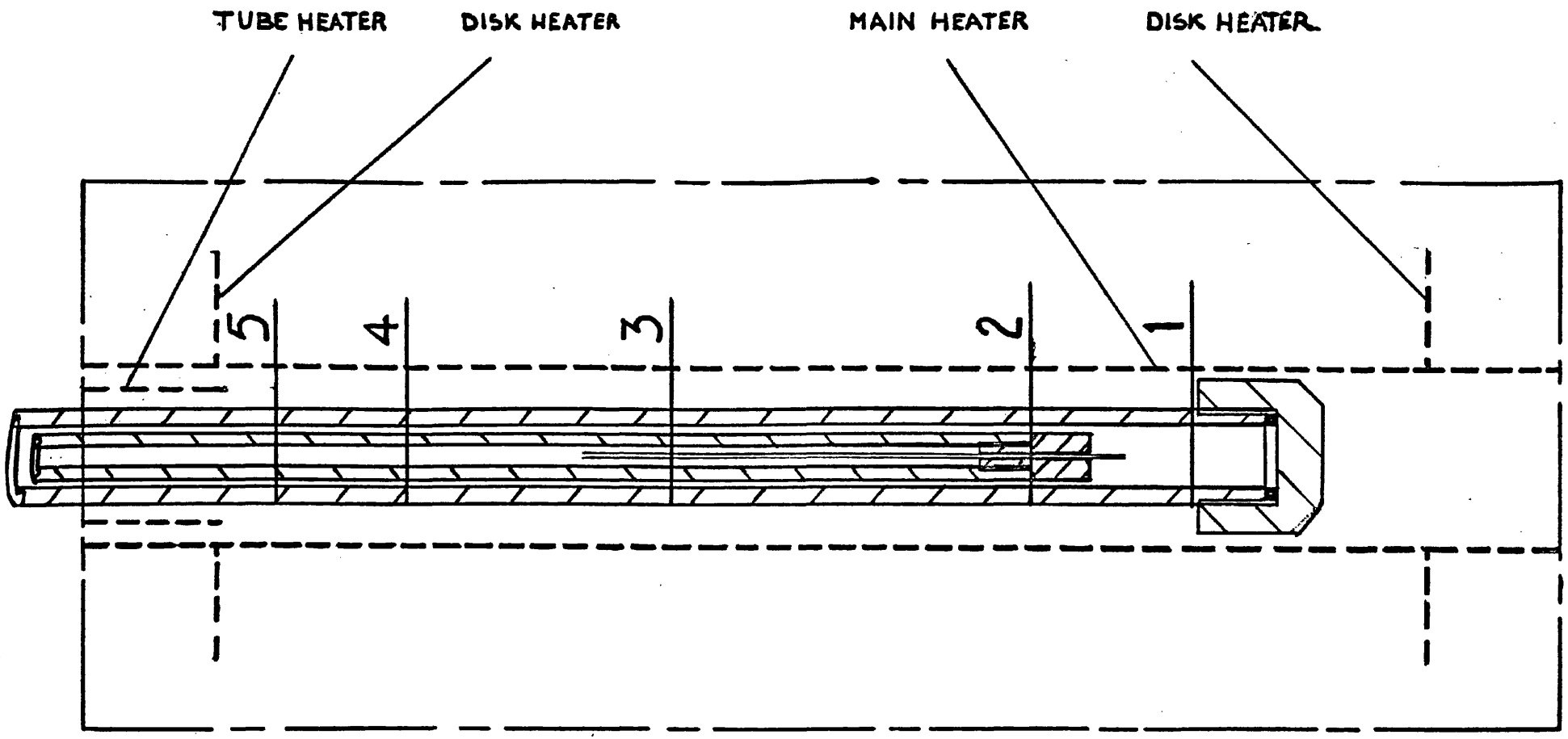
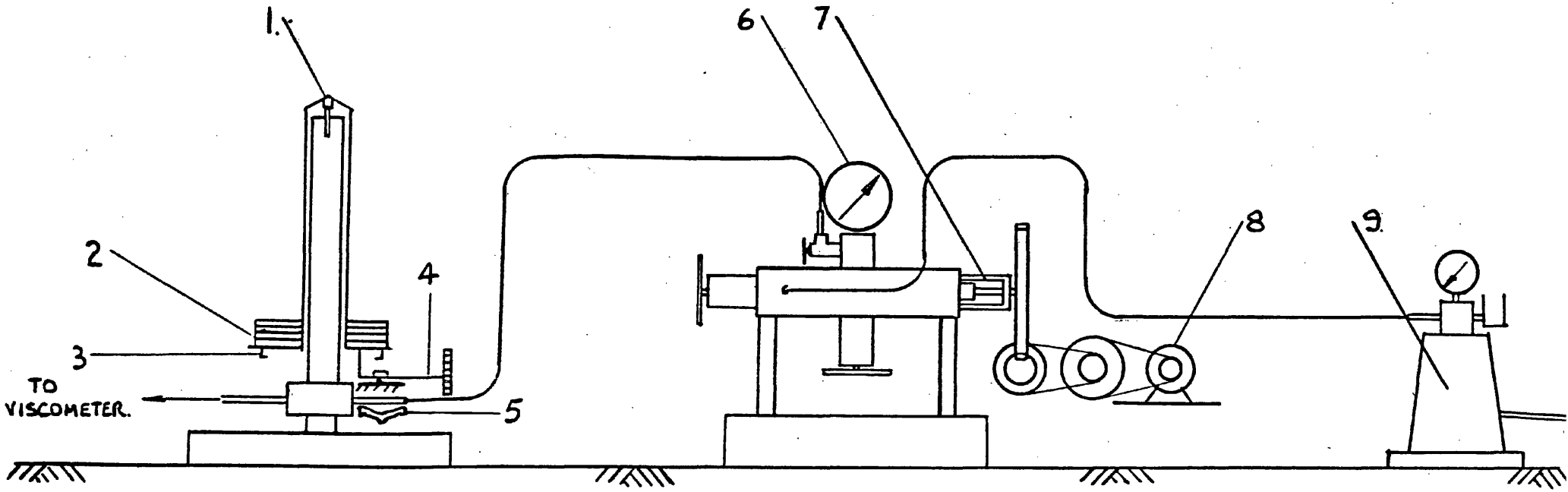


FIGURE 6

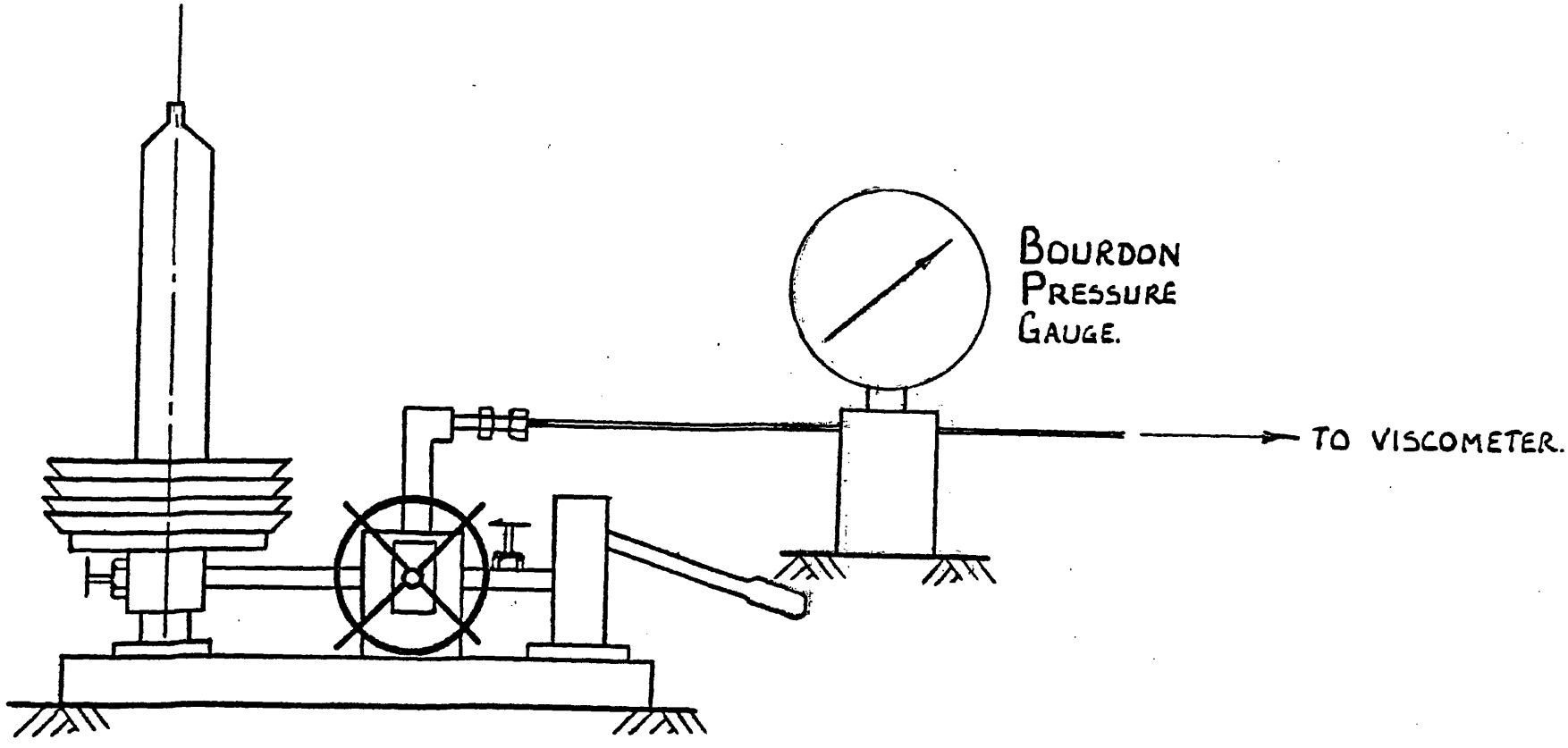
POSITION OF THERMOCOUPLES.



1. Pressure Balance Piston.
2. Overhung Weights.
3. Sprocket for Motor Drive.
4. Pointer Showing Piston Position
5. Mercury Switches to Actuate 8
6. Bourdon Pressure Gauge.
7. Ram Pump.
8. Motor.
9. Hand Pump.

FIGURE 7

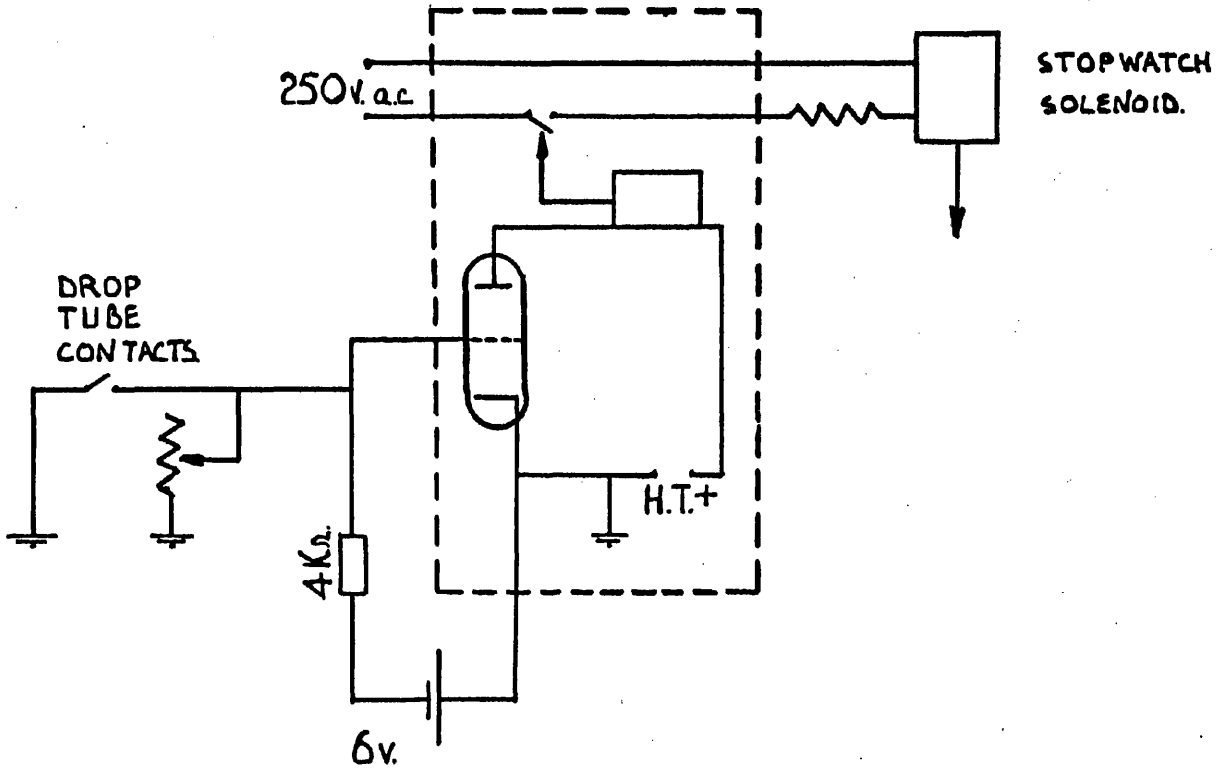
ORIGINAL PRESSURE SYSTEM.



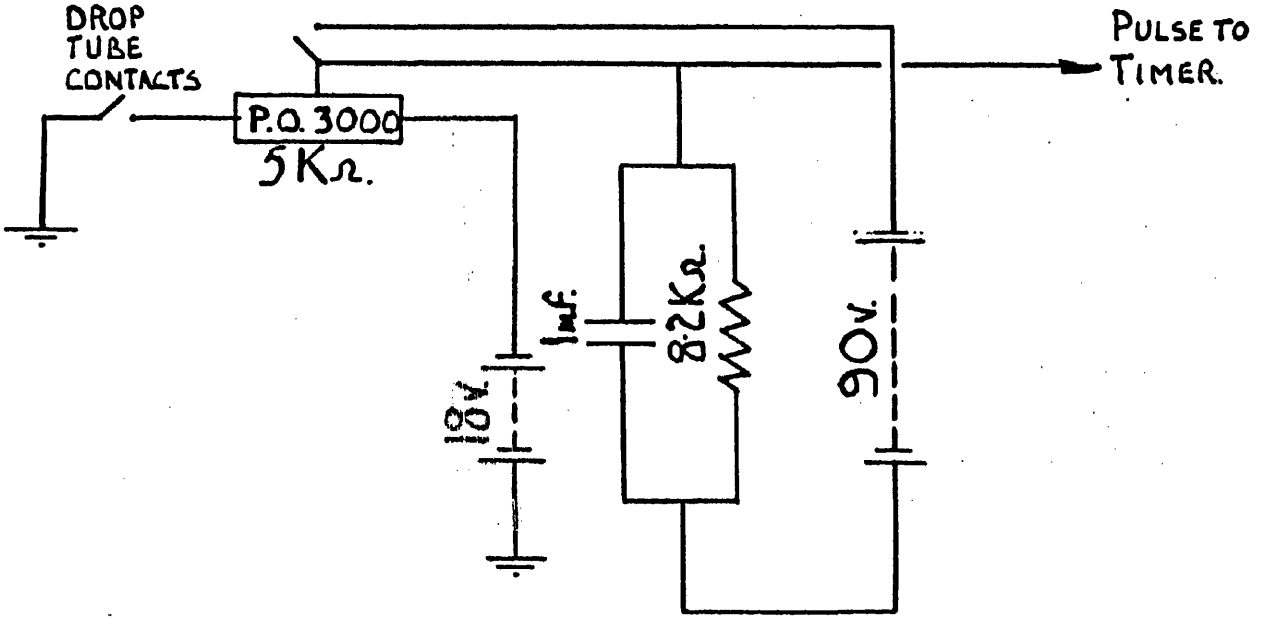
BUDENBERG DEAD-WEIGHT TESTER

FIGURE 8

PRESSURE SYSTEM.



TIMING CIRCUIT TO STOPWATCH.



INITIAL CALIBRATION TIMING CIRCUIT

FIGURE 9

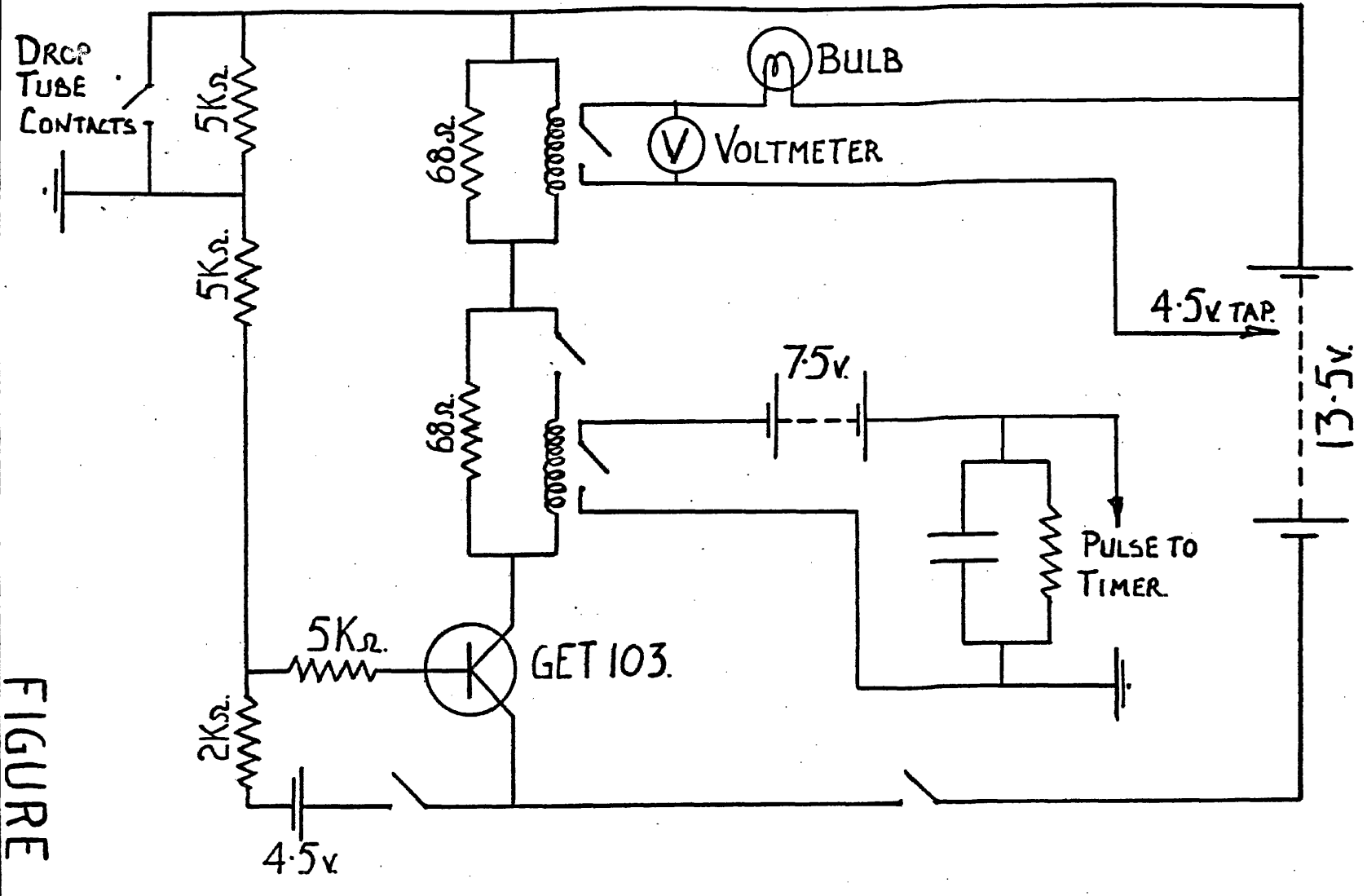


FIGURE 10

CALIBRATION TIMING CIRCUIT.

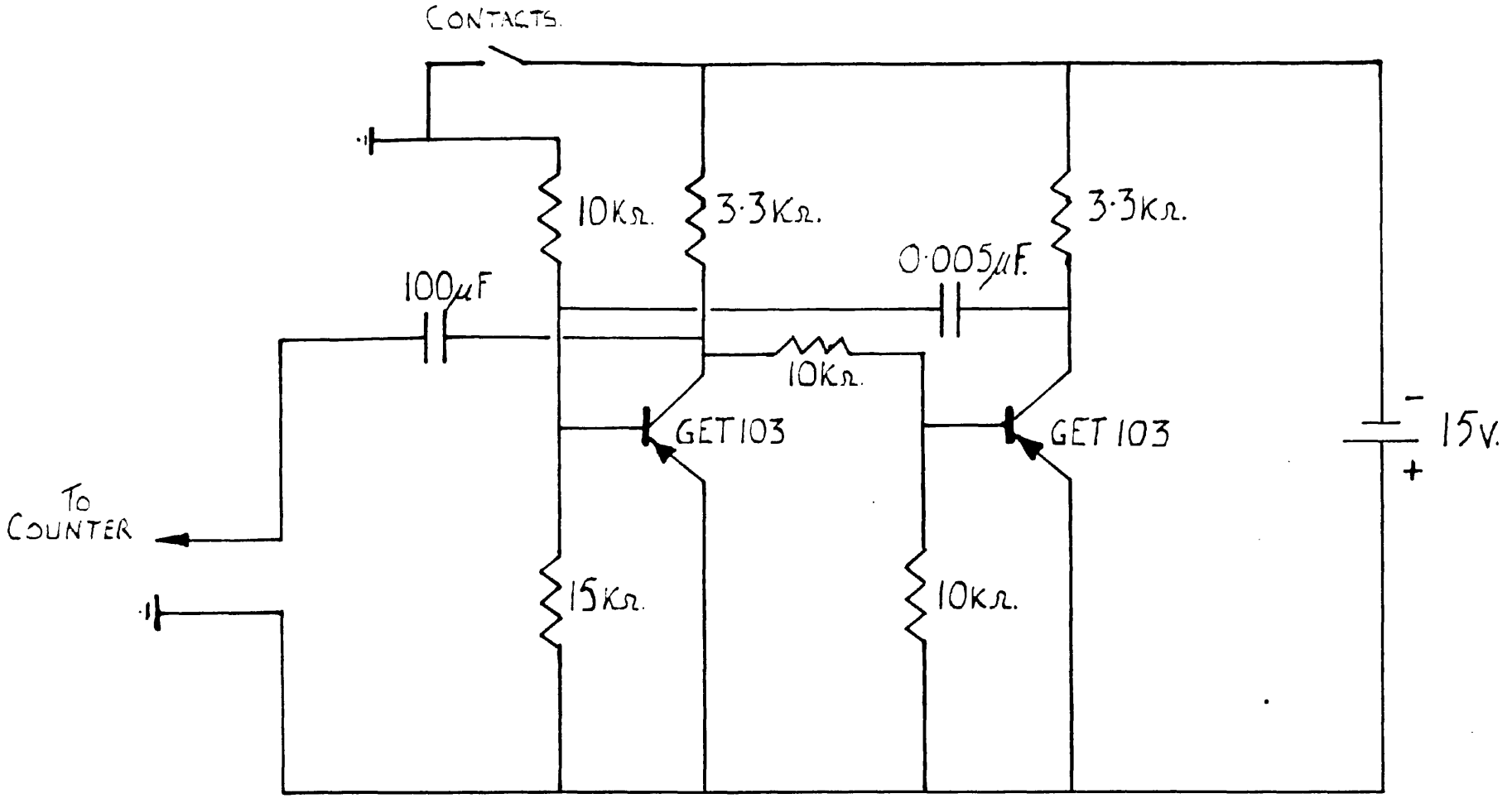


FIGURE 11

CALIBRATION TIMING CIRCUIT

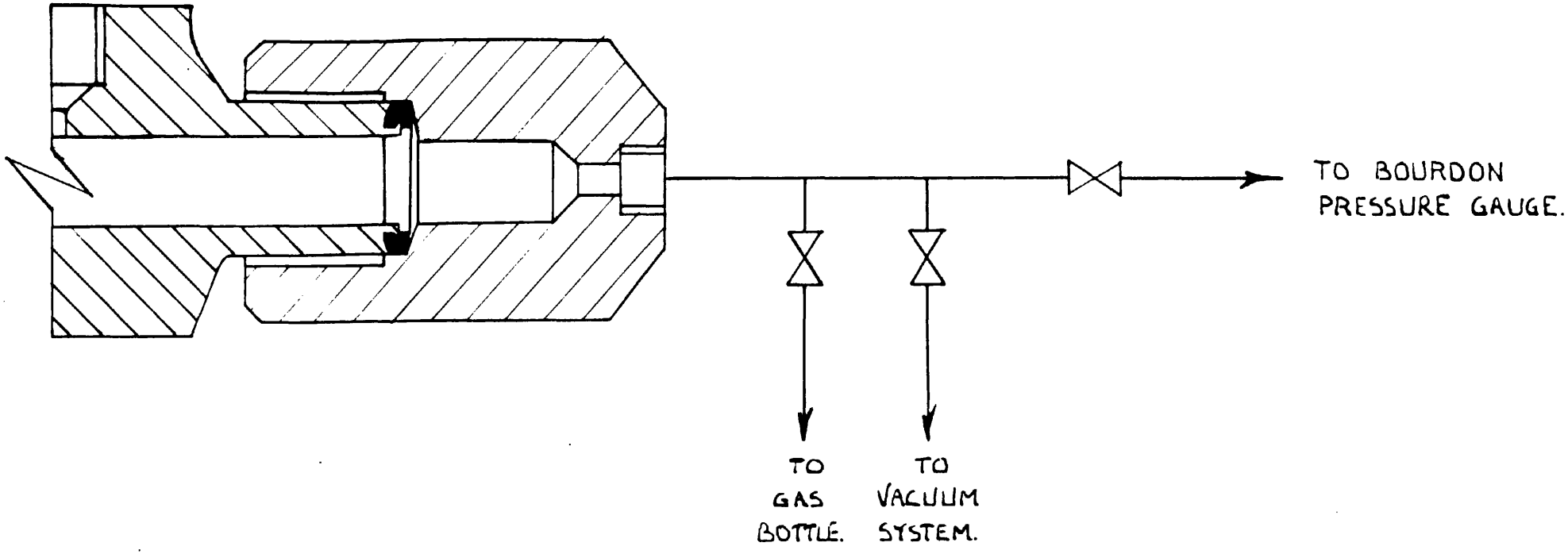


FIGURE 12.

PRESSURE RAISING & FILLING ARRANGEMENT FOR GASES.

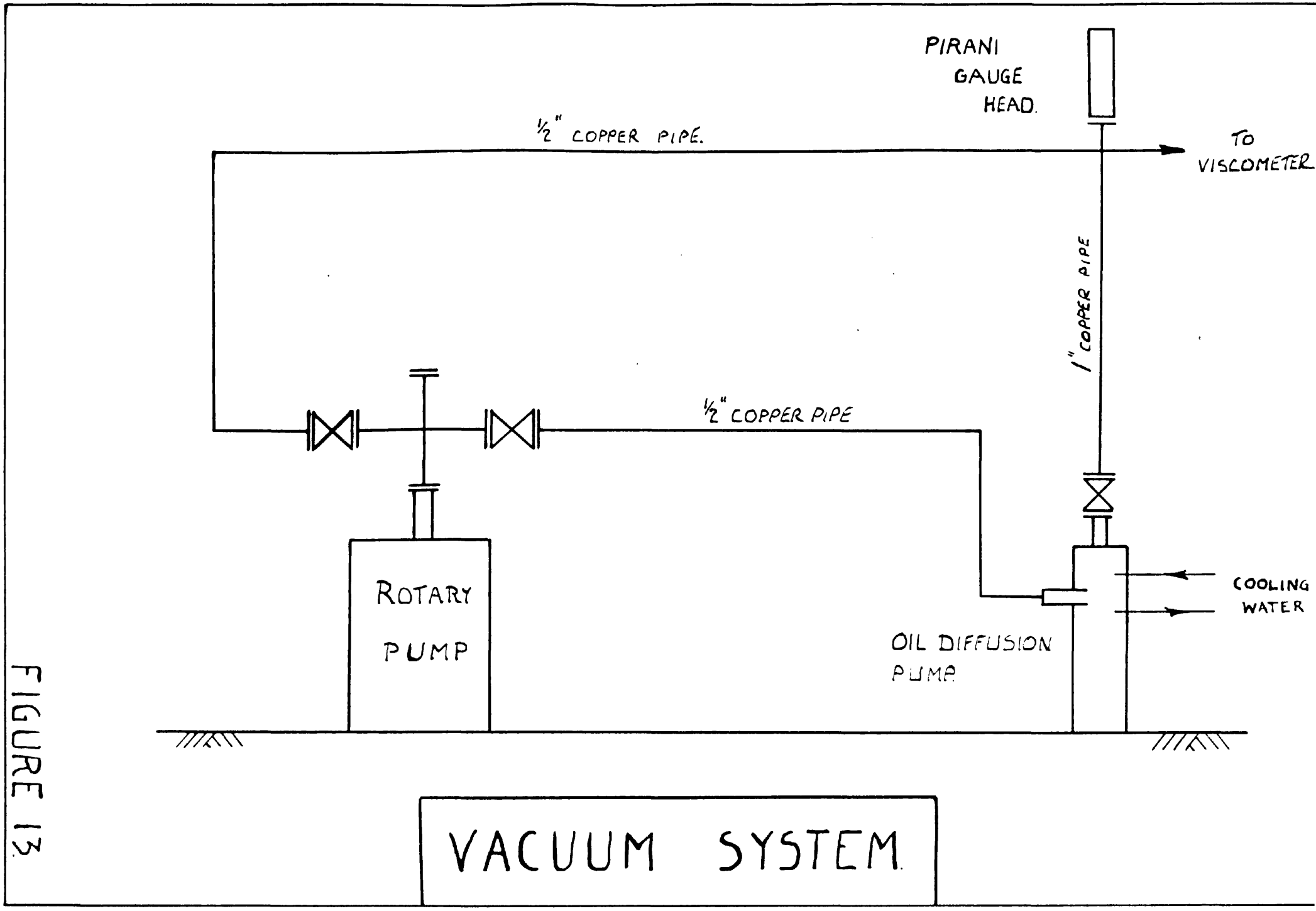


FIGURE 13.

VACUUM SYSTEM.

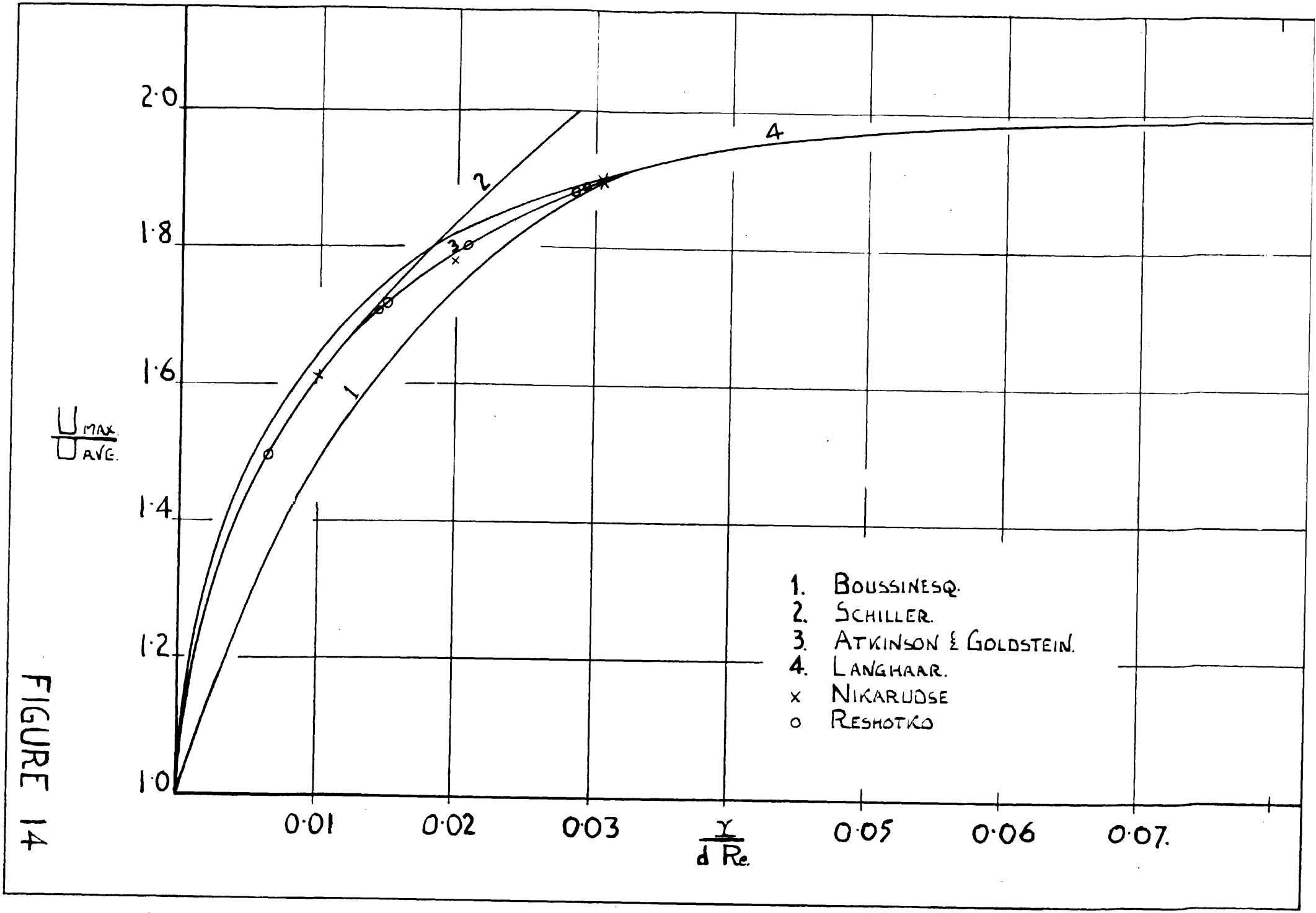

2·0
1·8
1·6
1·4
1·2
1·0
$\frac{U_{MAX.}}{U_{AVE.}}$
0·01
0·02
0·03
$\frac{y}{d\,Re}$
0·05
0·06
0·07.
1
2
3
4
1. Boussinesq.
2. Schiller.
3. Atkinson & Goldstein.
4. Langhaar.
x Nikaradse
o Reshotko

FIGURE 14

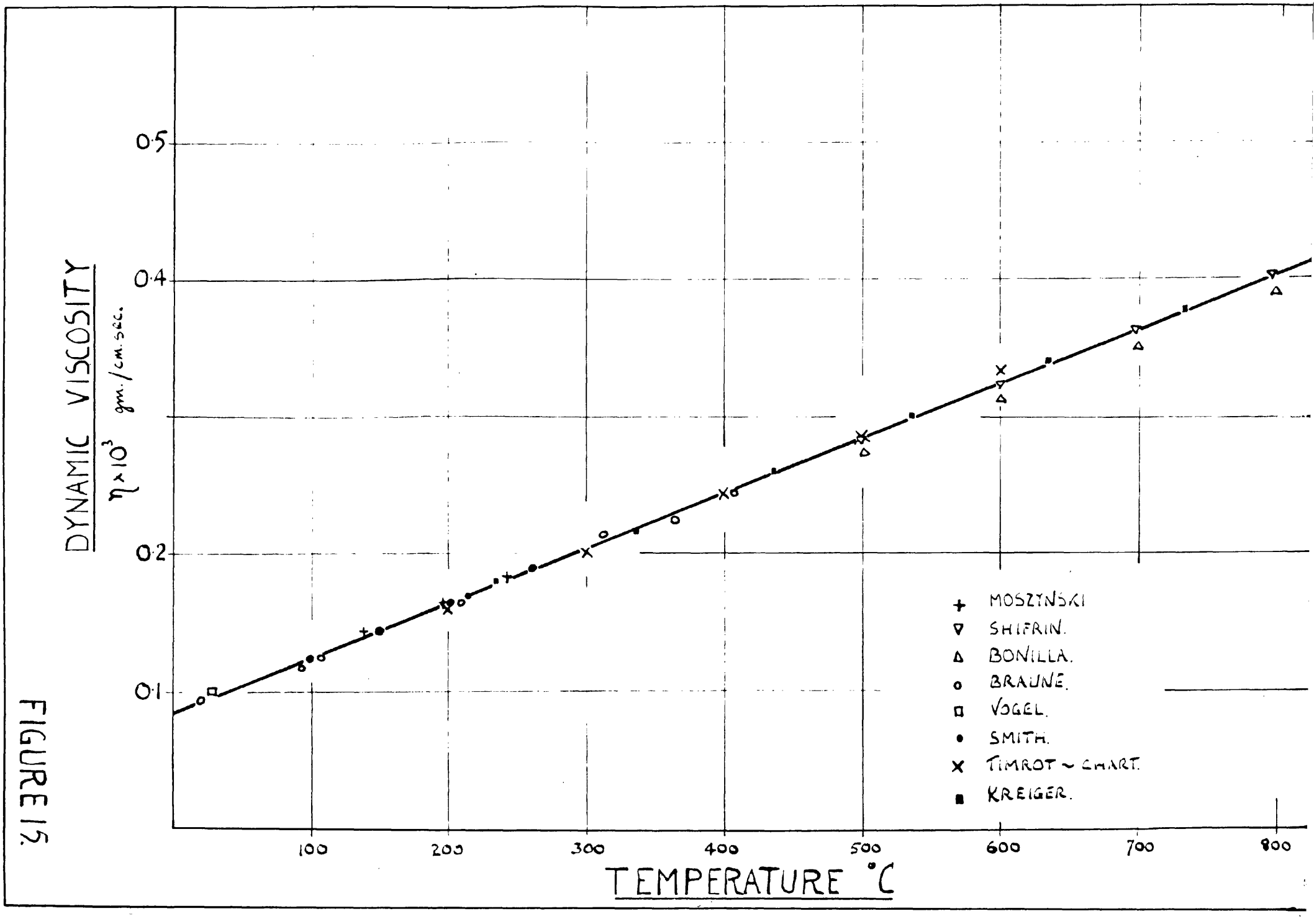

DYNAMIC VISCOSITY
$\eta \times 10^3$ gm./cm. sec.
0·5
0·4
0·2
0·1
100
200
300
400
500
600
700
800
TEMPERATURE °C
MOSZYNSKI
SHIFRIN.
BONILLA.
BRAUNE.
VOGEL.
SMITH.
TIMROT ~ CHART.
KREIGER.

FIGURE 7.

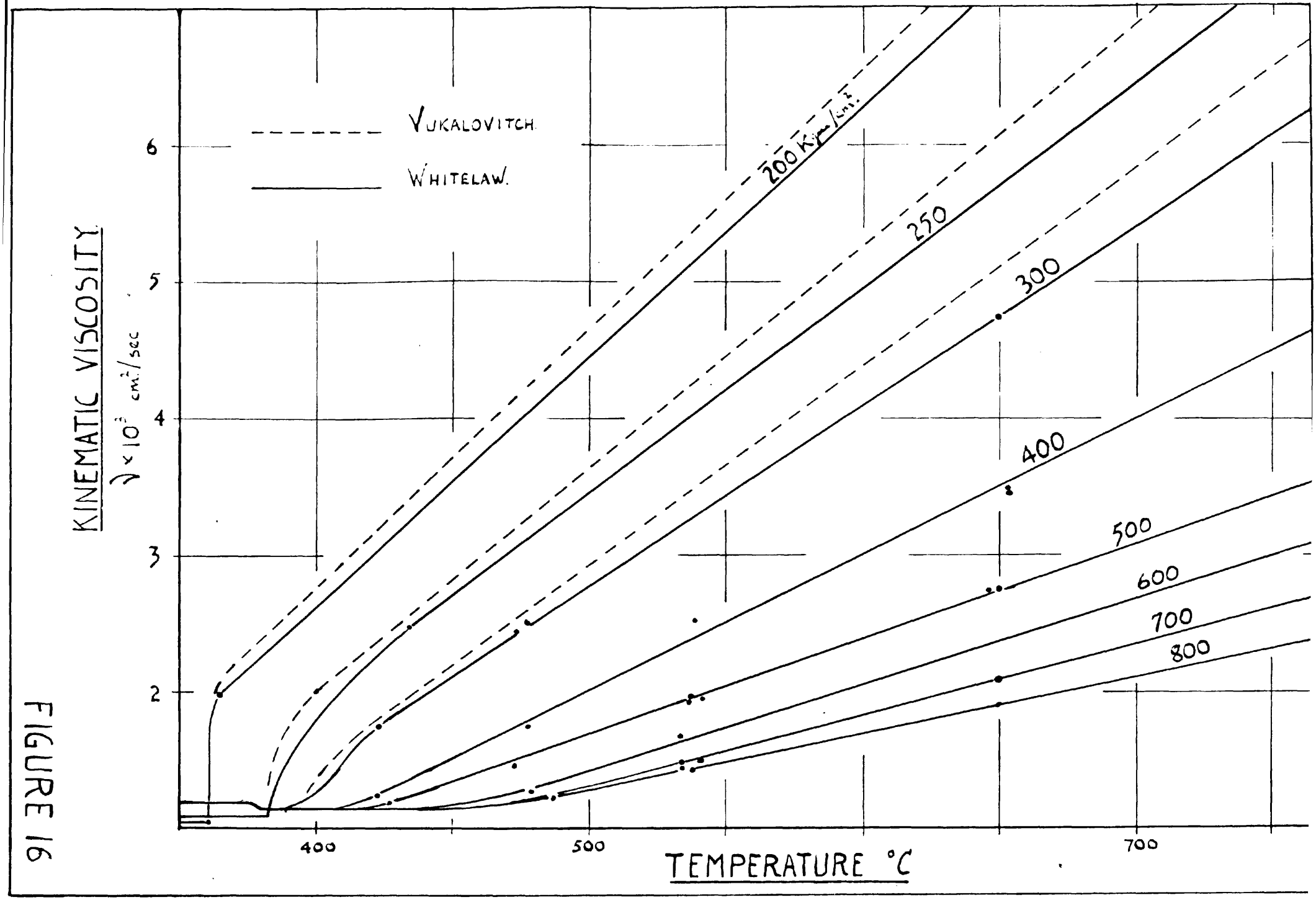

VUKALOVITCH
WHITELAW.
200 Kgm/cm²
250
300
400
500
600
700
800
KINEMATIC VISCOSITY
ν × 10³ cm²/sec
6
5
4
3
2
400
500
700
TEMPERATURE °C

FIGURE 16

DYNAMIC VISCOSITY

$\eta \times 10^3$ gm./cm. sec.

0·7

0·6

0·4

0·3

800 Kg/cm²

700

500

400

300

250

200

1 Kgm/cm².

400

500

TEMPERATURE °C

700

FIGURE 17

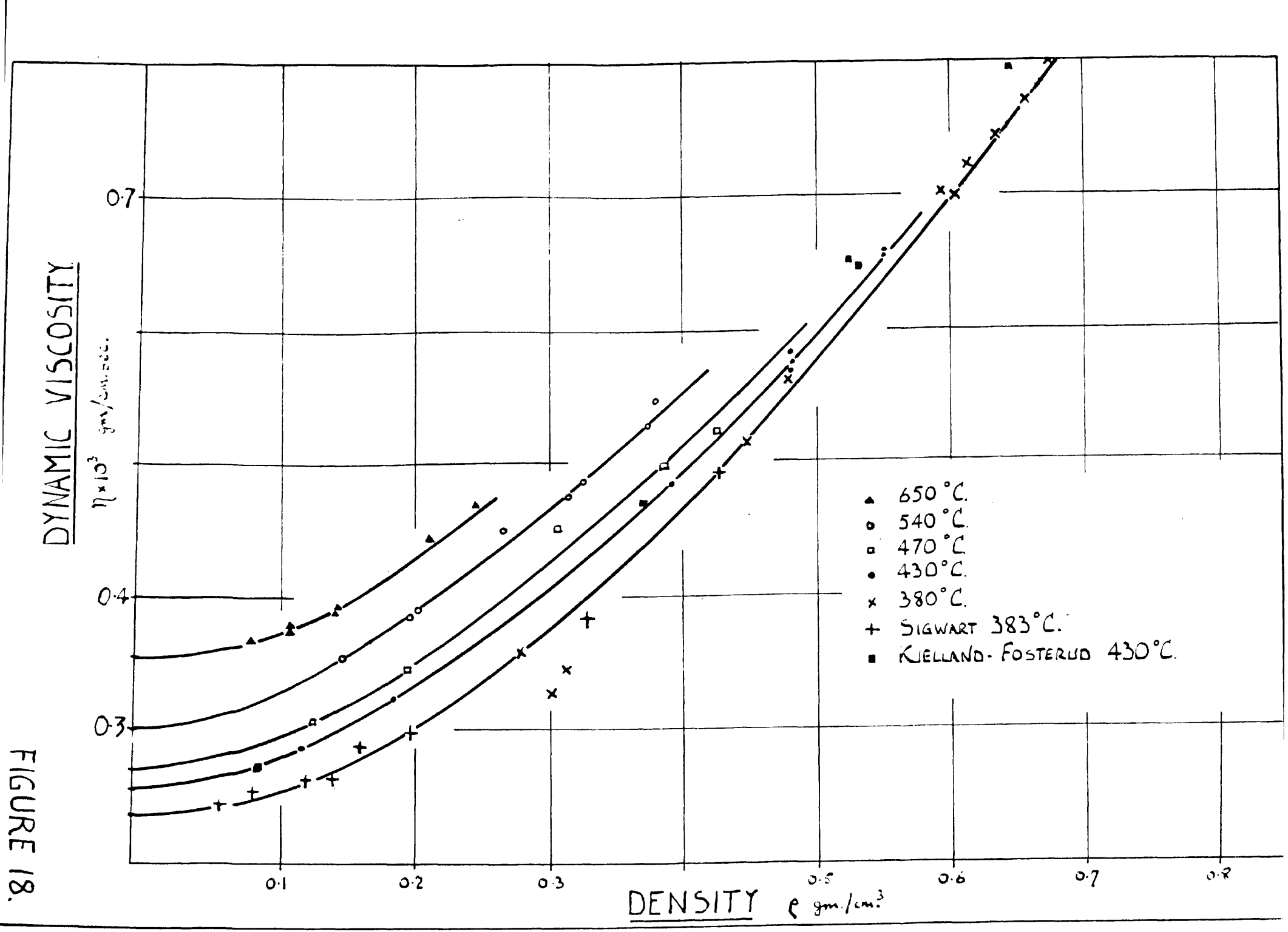

DYNAMIC VISCOSITY
η × 10³ gm/cm. sec.
0·7
0·4
0·3
0·1
0·2
0·3
0·5
0·6
0·7
0·8
DENSITY ρ gm./cm.³
650 °C.
540 °C.
470 °C.
430 °C.
380 °C.
SIGWART 383 °C.
KIELLAND-FOSTERUD 430 °C.

FIGURE 18.

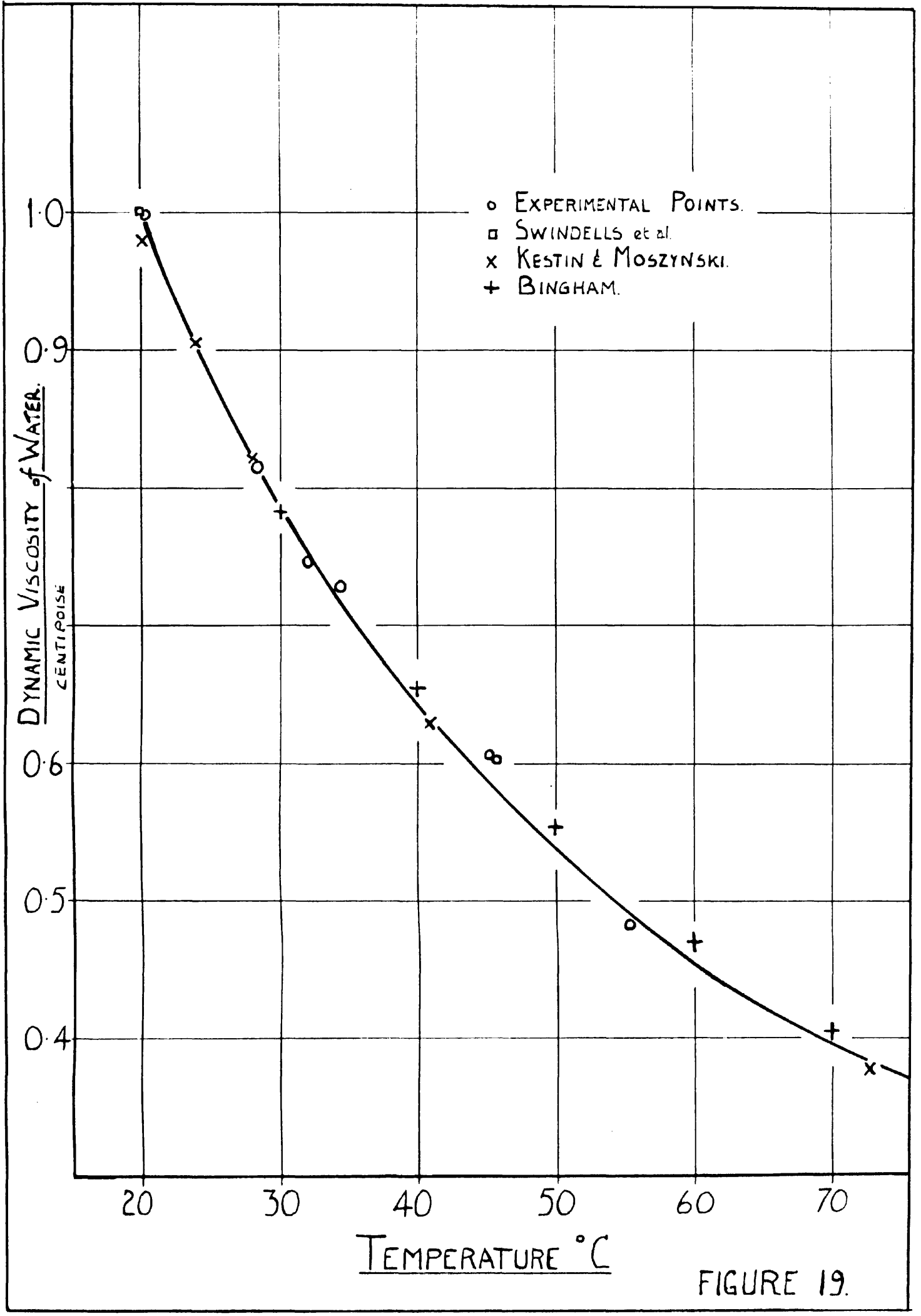

o EXPERIMENTAL POINTS.
□ SWINDELLS et al.
x KESTIN & MOSZYNSKI.
+ BINGHAM.
1·0
0·9
0·6
0·5
0·4
DYNAMIC VISCOSITY of WATER. CENTIPOISE
20
30
40
50
60
70
TEMPERATURE °C

FIGURE 19.

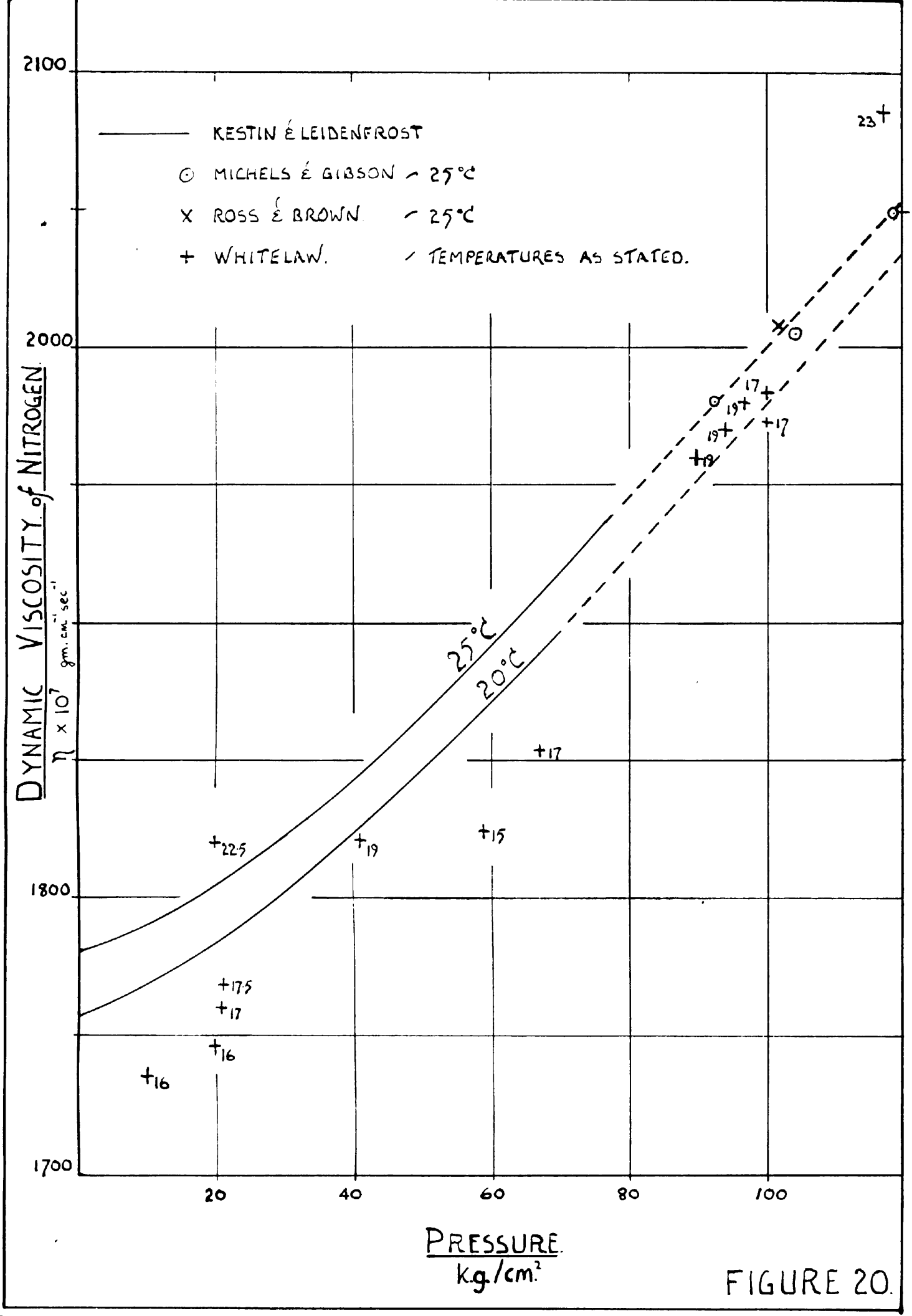
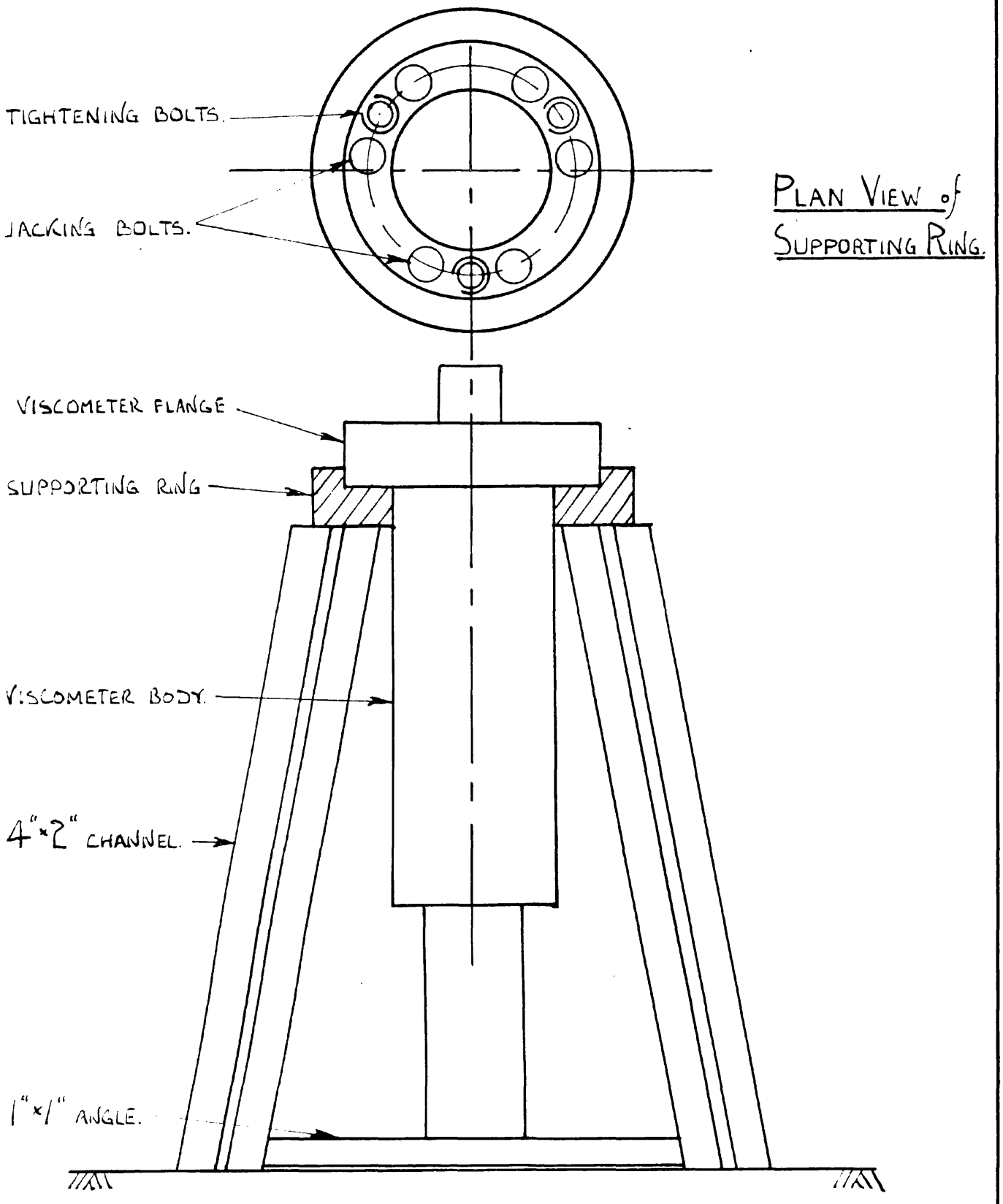

KESTIN & LEIDENFROST
MICHELS & GIBSON ~ 25°C
ROSS & BROWN ~ 25°C
WHITELAW. ~ TEMPERATURES AS STATED.
DYNAMIC VISCOSITY of NITROGEN
η x 10^7 gm. cm^-1 sec^-1
2100
2000
1800
1700
20
40
60
80
100
PRESSURE
kg/cm²
25°C
20°C
23
17
19
19
17
19
17
17
22·5
19
15
17·5
17
16
16

FIGURE 20.



PLAN VIEW of SUPPORTING RING.

FRAME.

FIGURE 21.

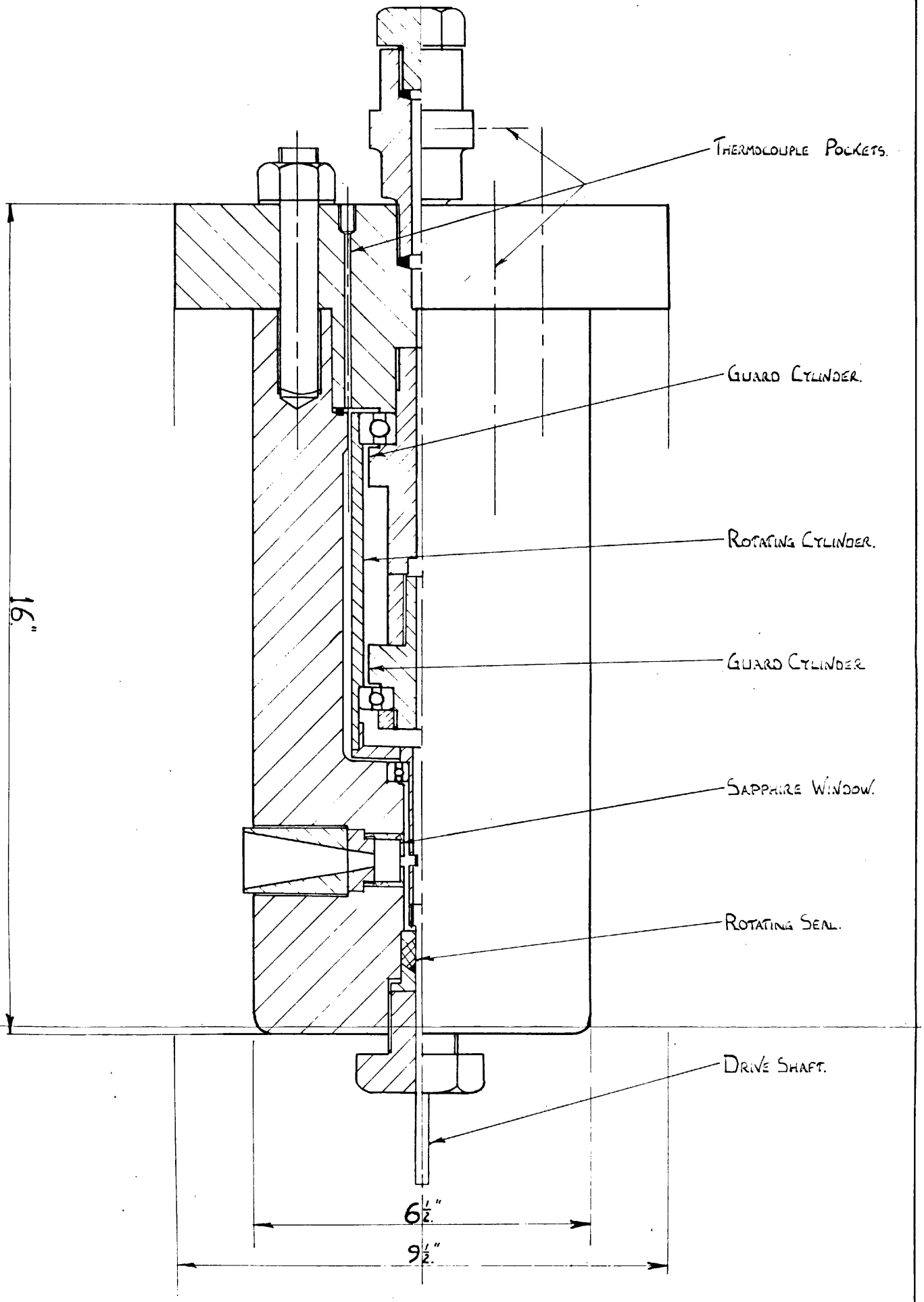
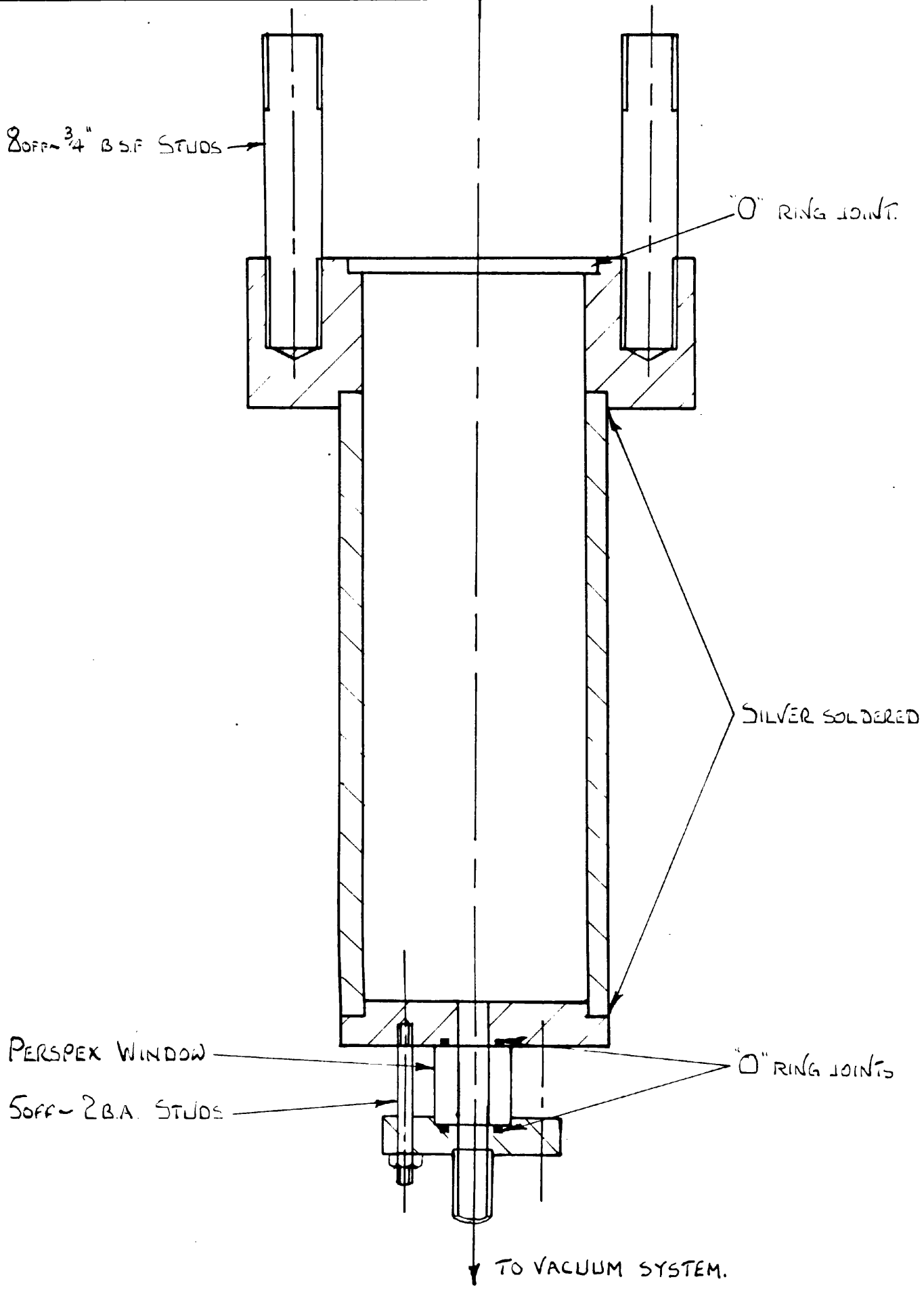


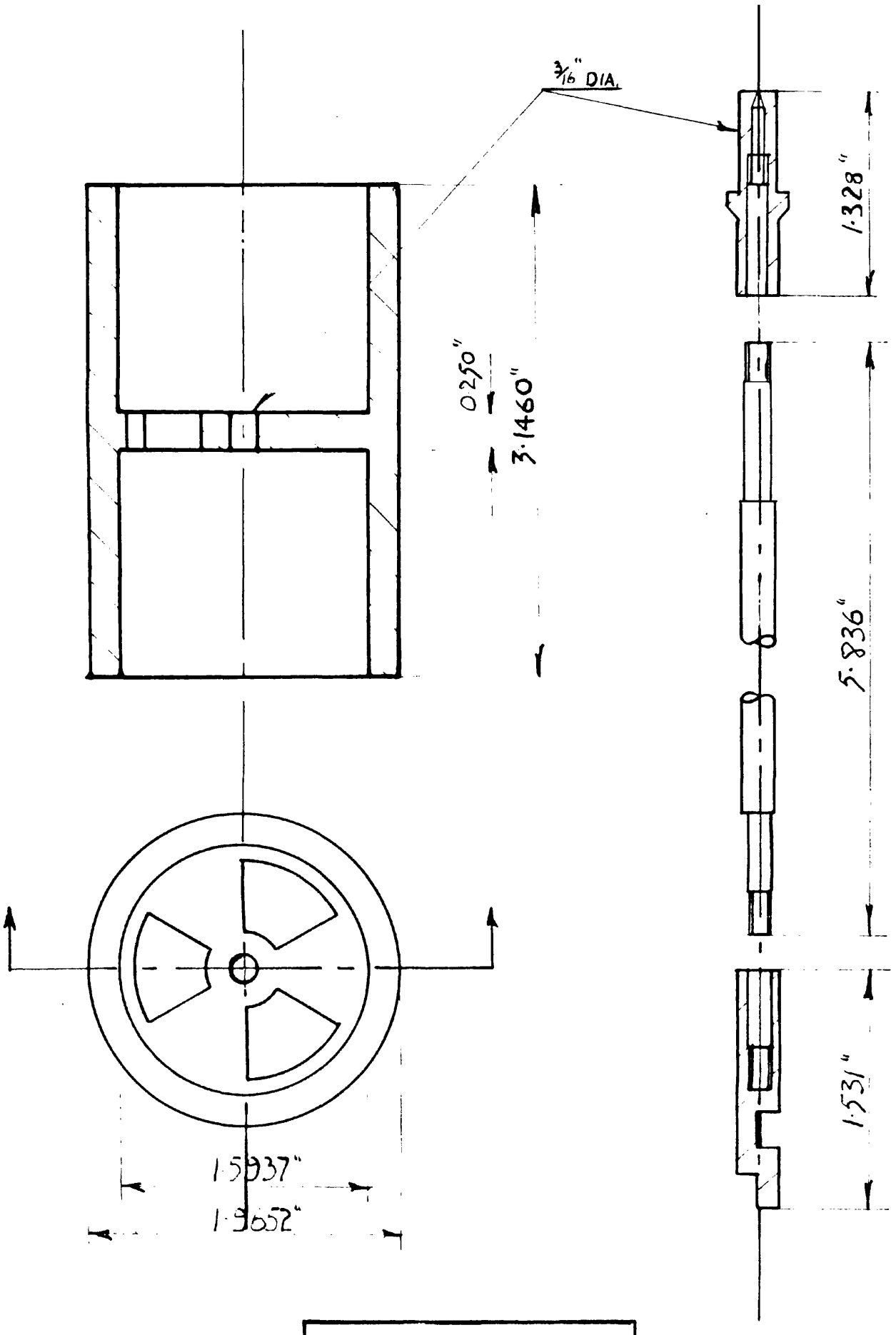
FIGURE 22

VISCOMETER ASSEMBLY



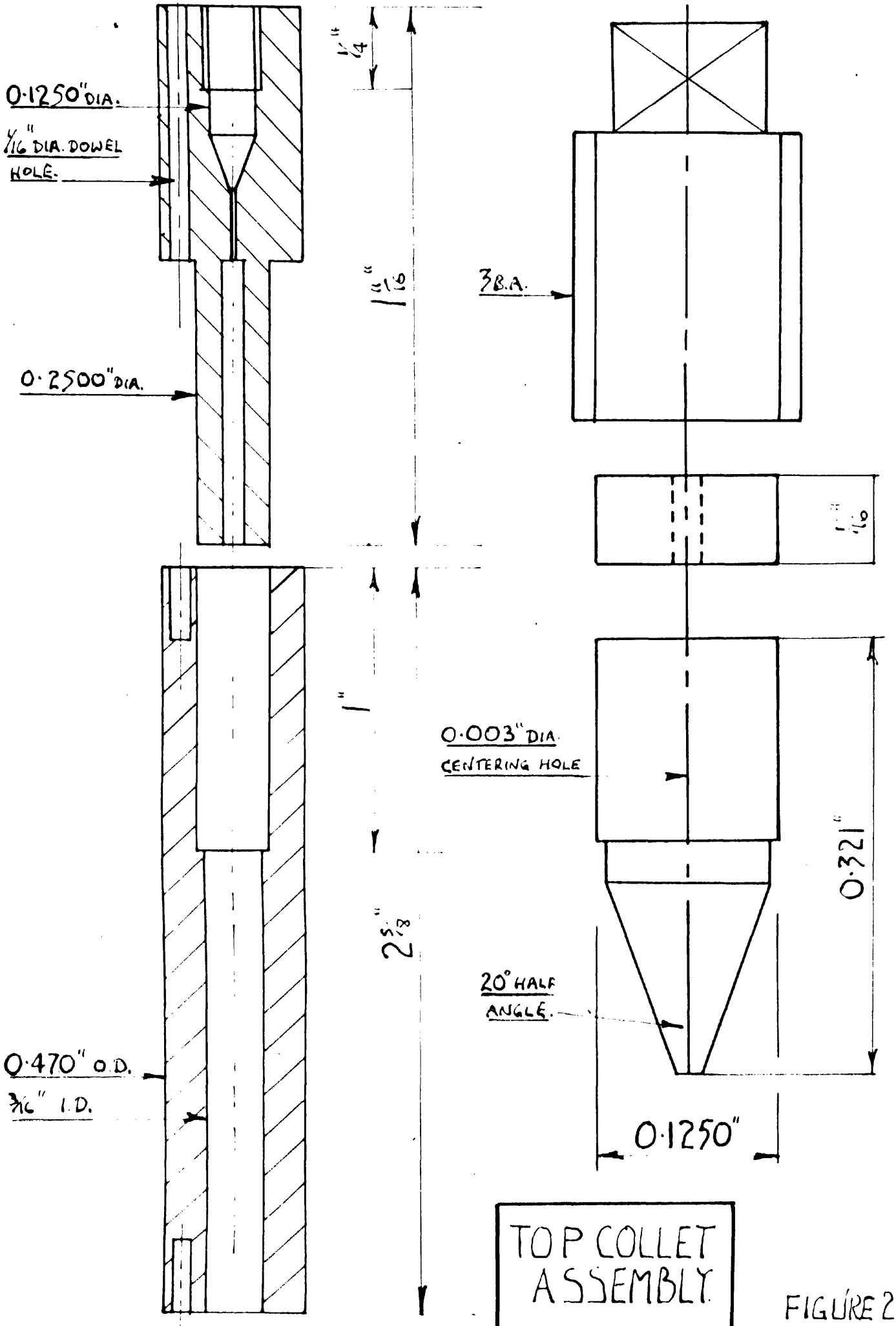
VACUUM VESSEL.

FIGURE 23.



SUSPENSION

FIGURE 24.



TOP COLLET ASSEMBLY

FIGURE 25

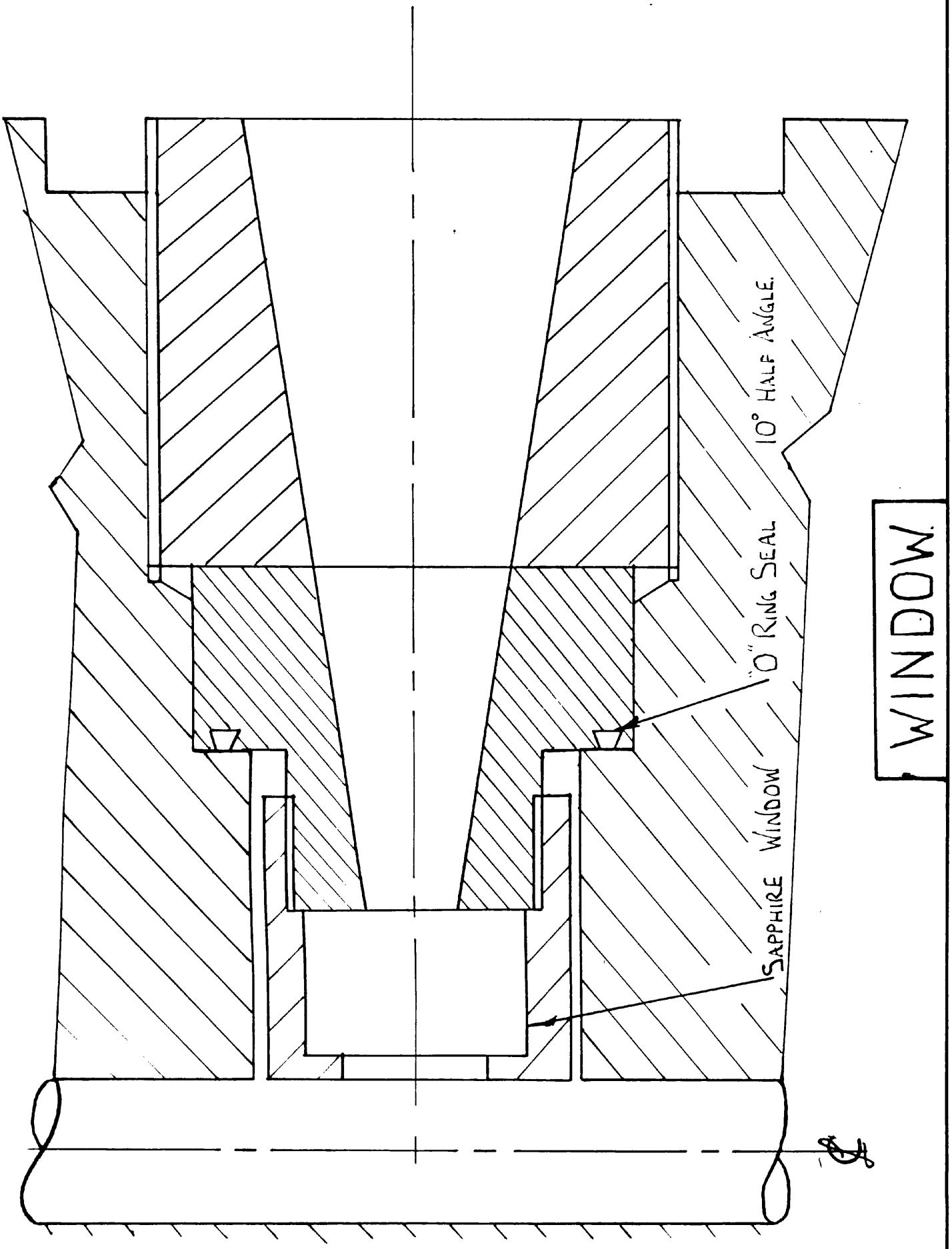
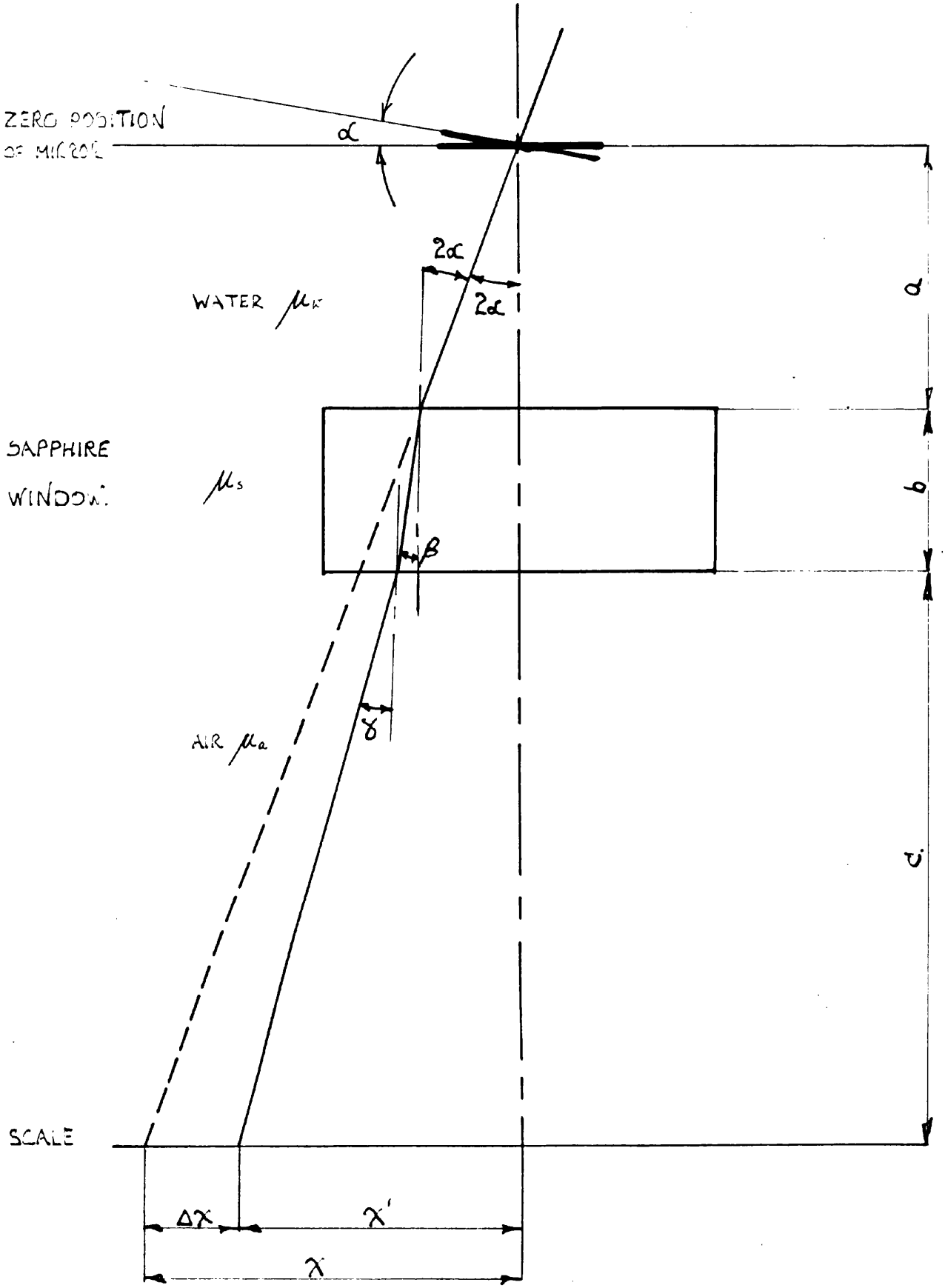


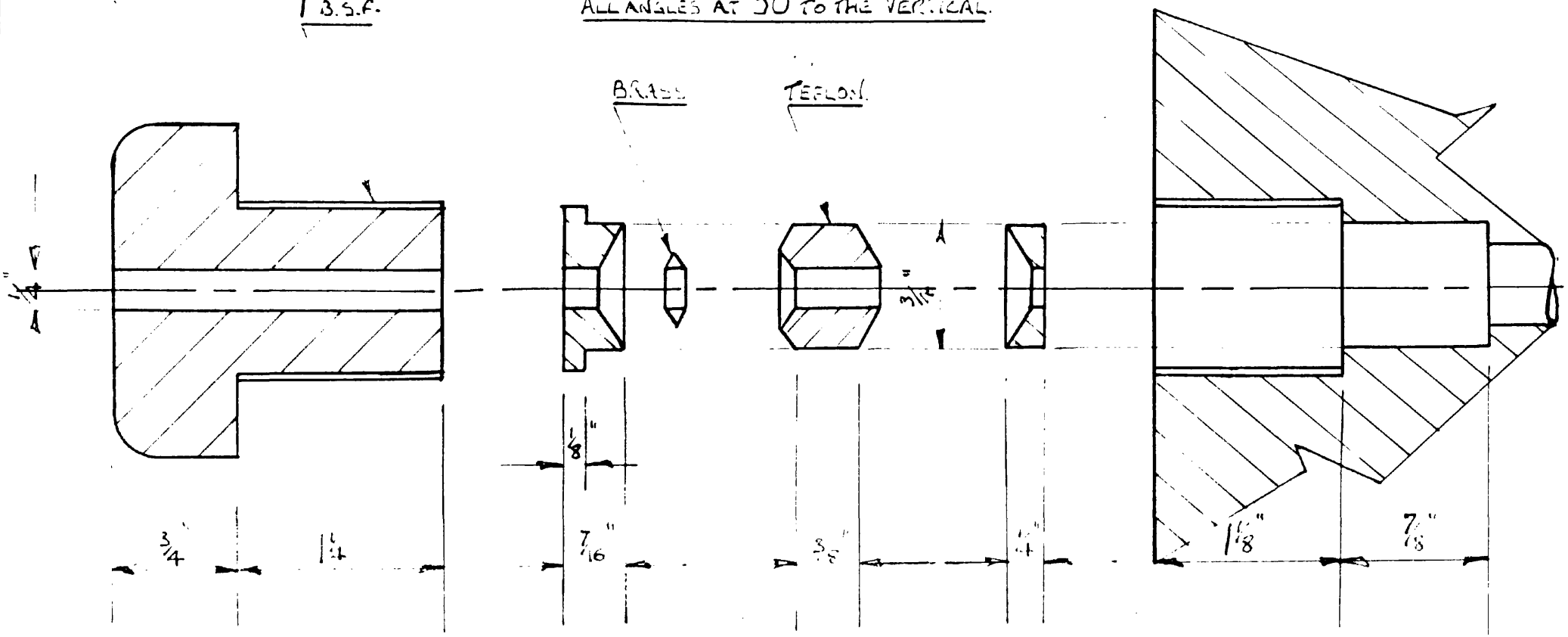
FIGURE 26



EFFECT OF REFRACTIVE INDEX. FIGURE 27.

1" B.S.F.

ALL ANGLES AT 30° TO THE VERTICAL.

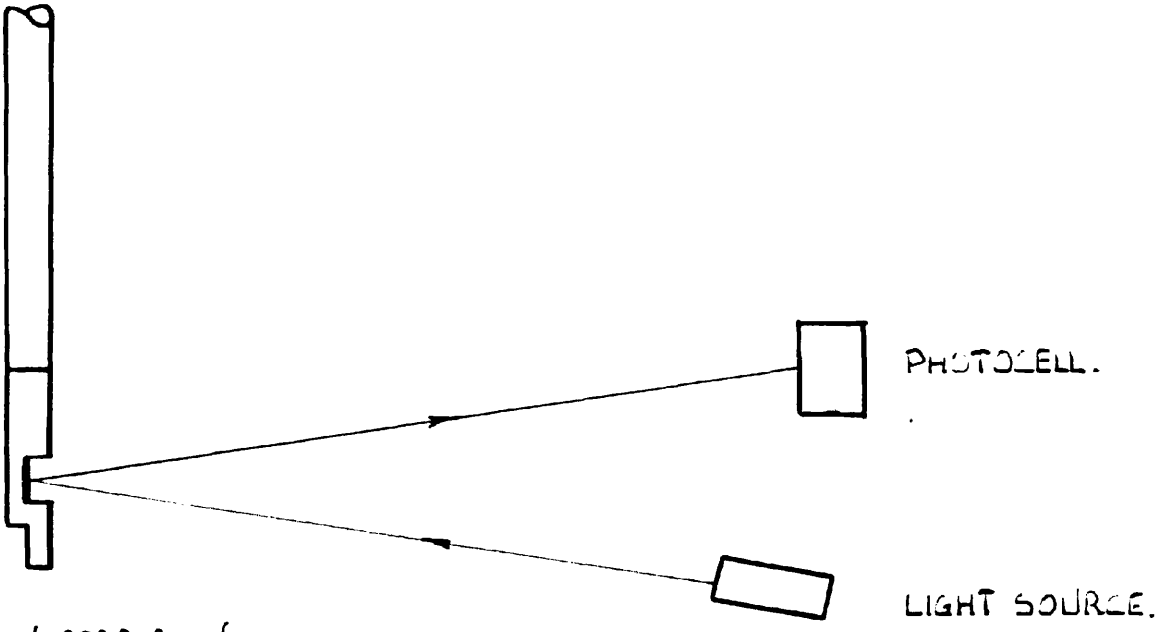


TEFLON PLUG TO BE DRIVING FIT ON 1/4" SHAFT & IN 3/4" HOLE.

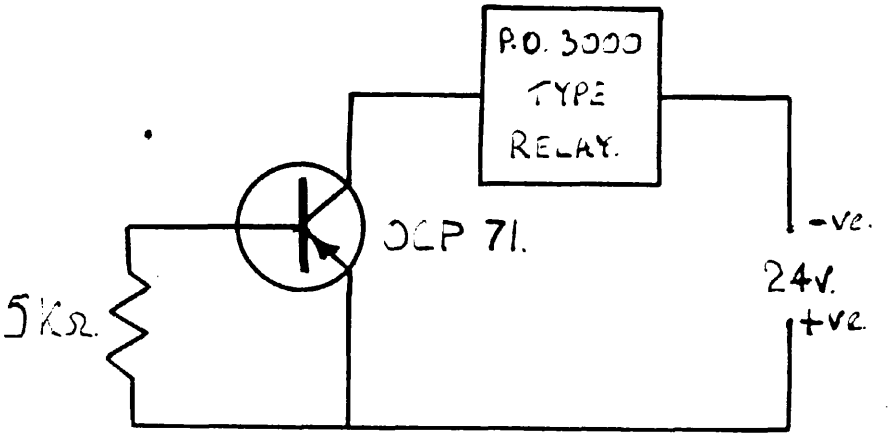
METAL PARTS OF SEAL TO BE SLIDING FITS.

FIGURE 28.

ROTATING SEAL.

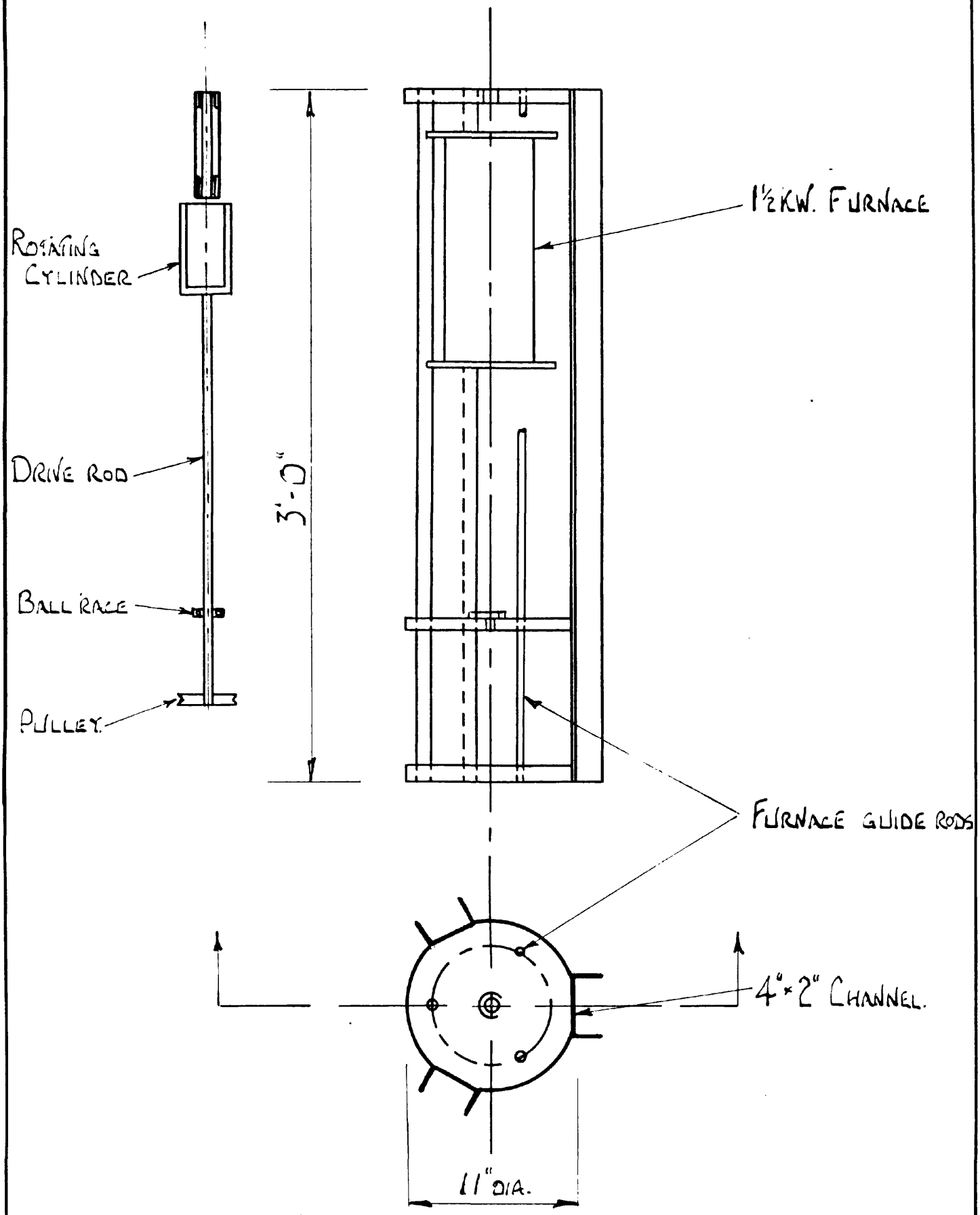


(a).



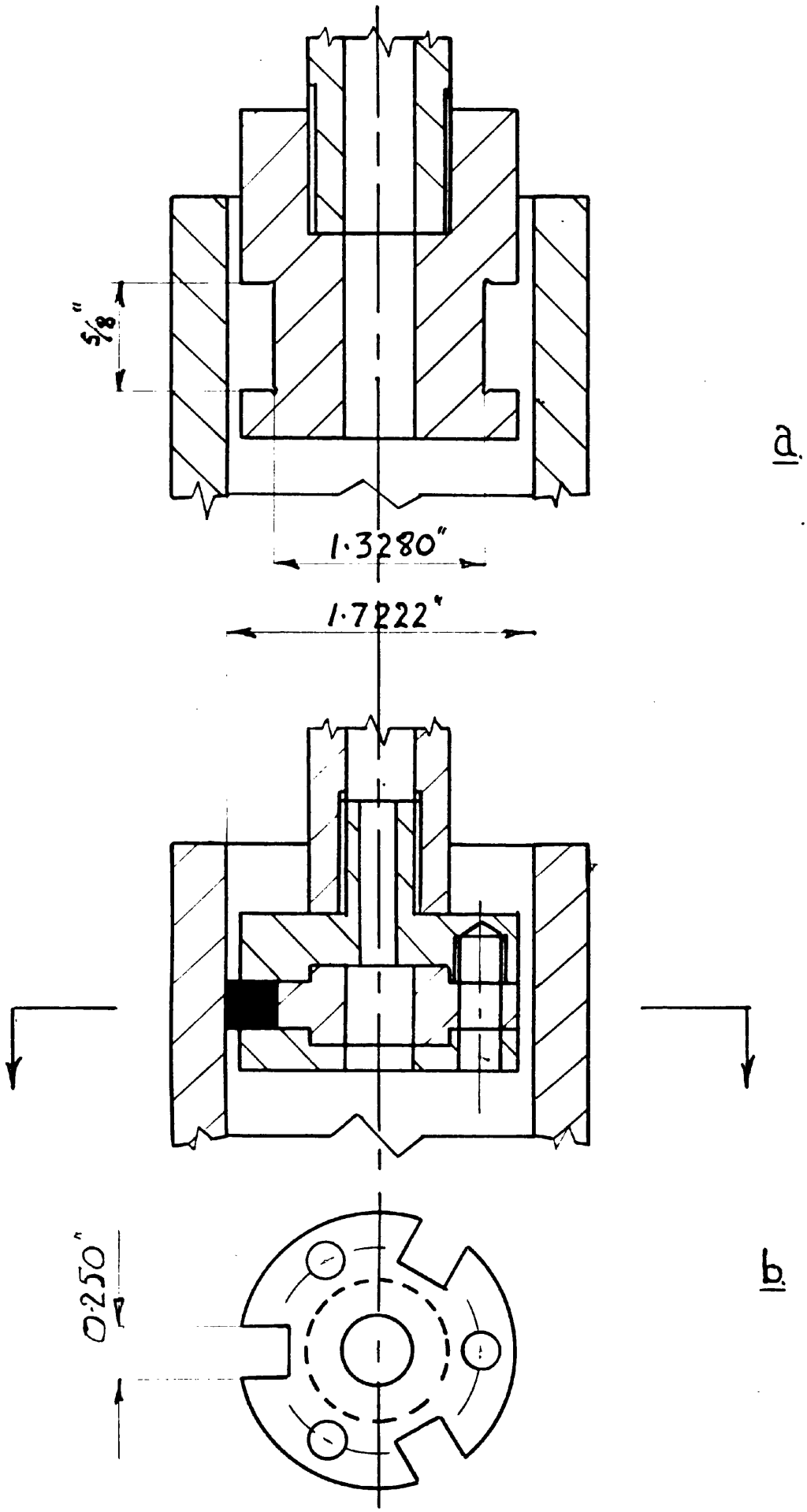
(b).

PERIOD MEASUREMENT FIGURE 29



BEARING TEST RIG.

FIGURE 30.



a.

b.

BEARING ASSEMBLIES.

FIGURE 31.